Contract No.: W912DQ-08-D-0018
Task Order No.: 014

U. S. Army Corps of Engineers
Kansas City District

Final Quality Assurance Project Plan Addendum No.7 Caged Bivalve Survey

Lower Passaic River Restoration Project

Remedial Investigation/Feasibility Study Oversight

Lower Passaic River Study Area, New Jersey

May 2, 2011

623558

CDM

14 Wall Street, Suite 1702
New York, New York 10005
tel: 212-377-4054
fax: 212-227-1692

May 2, 2011

United States Army Corps of Engineers
Kansas City District
601 East 12th Street, Room 463
Kansas City, Missouri 64106-2896

ATTN: CENWK-PM-ES/Buckrucker

CONTRACT: W912DQ-08-D-0018

PROJECT: Lower Passaic River Restoration Project
Remedial Investigation/Feasibility Study Oversight
Lower Passaic River Study Area, New Jersey

SUBJECT: Final Quality Assurance Project, Plan Addendum #7
Caged Bivalve Study

Dear Ms. Buckrucker:

CDM Federal Programs Corporation (CDM) is pleased to submit this electronic copy of the Final Quality Assurance Project Plan, Addendum No. 7 for the Oversight of Remedial Investigation/Feasibility Caged Bivalve Study in support of the Lower Passaic River Restoration Project in the Lower Passaic River Study Area, New Jersey. This document is based on the CPG's Benthic QAPP Addendum Number 4.

If you have any comments concerning this submittal, please contact me at (212) 377-4056.

Very truly yours,
CDM FEDERAL PROGRAMS CORPORATION

Frank Tsang, P.E.
Project Manager

Attachment
cc: S. Vaughn, EPA
J. Czapor, CDM (Letter Only)
G. Molnar, CDM
Project File
Bill Sy, EPA
S. Budney, CHMM, CDM
J. Oxford, CHMM, CDM

LOWER PASSAIC RIVER RESTORATION PROJECT
OPERABLE UNIT (OU) 2

Remedial Investigation/Feasibility Study Oversight

Final Quality Assurance Project Plan

Addendum No.7

Caged Bivalve Study

Lower Passaic River Study Area, New Jersey

USACE CONTRACT No. W912DQ-08-D-0018

TASK ORDER No. 014

May 2, 2011

Prepared for:
U.S. Army Corps of Engineers
Kansas City District

Prepared by:
CDM
110 Fieldcrest Avenue, 6th Floor
Raritan Center
Edison, New Jersey 08837

TABLE OF CONTENTS

Notes:
1. Appendix K is included in the Oversight QAPP, Addendum No. 5, Fish Tissue Analysis

Worksheets not included herein are included in the

- Physical Water Column Monitoring/Generic Final QAPP dated March 9, 2010 and the
- Final QAPP Addendum Number 5, Fish Tissue Analysis, dated August 2010.

Acronyms

%	percent
%D	percent difference
%R	percent recovery
µg/g	microgram per gram
µg/L	microgram per liter
A	analytical
AAS	atomic absorption spectrometry
ABS	absolute difference
AES	atomic emission spectrophotometry
ANSETS	Analytical Services Tracking System
ASC	analytical services coordinator
ASTM	American Society of Testing and Materials
BS	Bachelor of Science
CA	corrective action
CAS	Chemical Abstract Service
CCV	continuing calibration verification
CD	compact disk
CDM	Camp, Dresser & McKee
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CHMM	Certified Hazardous Materials Manager
CIH	certified industrial hygienist
CLP	Contract Laboratory Program
COC	chain of custody
CPG	Cooperating Parties Group
CRM	certified reference material
CRQL	contract required quantitation limits
CVAFS	cold vapor atomic fluorescence spectrometry
DESA	Division of Environmental Science and Assessment
DL	detection limit
DoD	Department of Defense
DQA	data quality assessment
DQI	data quality indicators
DQL	data quality level
DQO	data quality objectives
DV	data validation
EDD	electronic data deliverable
EPA	United States Environmental Protection Agency

EQL	estimated quantitation limit
ESAT	EPA data validation contractor
FAR	Federal Acquisition Regulations
FASTAC	Field and Analytical Services Teaming Advisory Committee
FID	flame ionization detector
FS	feasibility study
FSP	field sampling plan
FTL	field task leader
GC/MS	gas chromatograph / mass spectroscopy
H&S	health and safety
H&SM	health and safety site manager
HASP	Health and Safety Plan
HDPE	high density polyethylene
HPLC	High Pressure Liquid Chromatography
HQ	headquarters
HRGC/HRMS	High Resolution Gas Chromatography / High Resolution Mass Spectrometry
HRGC/LRMS	High Resolution Gas Chromatography / Low Resolution Mass Spectrometry
ICAL	initial calibration
ICP	inductively coupled plasma
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectrometry
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
ID	identification
IPR	initial precision and recovery
IR	infra-red
KC	Kansas City
LAN	local area network
LC	lethal concentration
LCS	laboratory control samples
LCSD	laboratory control sample duplicates
LPR	Lower Passaic River
Ltd.	limited
MDL	method detection limit
mg/kg	milligram per kilogram
mg/L	milligram per liter
MPI	Malcolm Pirnie Inc.
MS	matrix spike
MS/ MSD	matrix spikes /matrix spike duplicate
NA	not available or not applicable

ng/g	nanogram per gram
ng/kg	nanogram per kilogram
NJ	New Jersey
NJDEP	New Jersey Department of Environmental Protection
NJDOT	New Jersey Department of Transportation
NOAA	National Oceanic Atmospheric Administration
NY	New York
°C	degrees Celsius
OPR	ongoing precision and recovery
OU	operable unit
oz	ounce
PAH	polycyclic aromatic hydrocarbon
PAL	project action limit
PCB	polychlorinated biphenyl
PCDD/PCDF	polychlorodibenzodioxin /polychlorodibenzofurans
pg/g	picogram per gram
PM	project manager
PPE	Personal Protection Equipment
ppt	parts per thousand (salinity unit)
PQL	project quantitation limit
PQLG	project quantitation limit goal
PQO	project quality objective
PREmis	Passaic River Estuary Management Information System
PRP	potentially responsible party
PT	Performance Test
PWCM	Physical Water Column Monitoring
QA	quality assurance
QAC	quality assurance coordinator
QAPP	quality assurance project plan
QC	quality control
QCS	quality control sample
QL	quantitation limit
QP	quality procedure
RA	remedial action
RAS	routine analytical services
RI/FS	Remedial Investigation / Feasibility Study
RPD	relative percent difference
RPM	remedial project manager

RSCC	Regional Sample Control Coordinator
RSD	relative standard deviation
S&A	sampling and analytical
SA	self assessment
SDG	Sample Delivery Group
SIM	selective ion monitoring
SM	Standard Method
SOP	standard operating procedure
SOW	scope of work
SVOC	semivolatile organic compound
TAL	target analyte list
TBD	to be determined
TCL	target compound list
TSOP	Technical Standard Operating Procedure
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service
WS	worksheet

Dioxin and Furans:

HpCDD	hepta-chlorodibenzo-*p*-dioxin
HpCDF	hepta-chlorodibenzofuran
HxCDD	hexa-chlorodibenzo-*p*-dioxin
HxCDF	hexa-chlorodibenzofuran
OCDD	octa-chlorodibenzo-*p*-dioxin
OCDF	octa-chlorodibenzofuran
PeCDD	penta-chlorodibenzo-*p*-dioxin
PeCDF	penta-chlorodibenzo-furan
TCDD	tetrachloro-dibenzo-*p*-dioxin
TCDF	tetrachloro-dibenzo-furan

Introduction

CDM Federal Programs Corporation (CDM) will accept split caged bivalve tissue samples from the Cooperating Parties Group (CPG) upon completion of the in situ caged bivalve study in the Lower Passaic River Study Area (LPRSA).

This Final Quality Assurance Project Plan (QAPP) Addendum No.7 and the *Lower Passaic River RI/FS Oversight Final QAPP, Physical Water Column Monitoring and Generic Information for Upcoming Tasks*, dated March 2010 (hereafter referred to as the Final QAPP) are the governing documents for execution of this analytical investigation. CDM will use the various plans prepared by the CPG contractors to verify proper execution of the bivalve tissue sample handling, preservation and shipment.

The Final QAPP indicated that future oversight tasks assigned to CDM would be appended with selected worksheets. The following worksheets are included in this addendum to reflect only the caged bivalve tissue analytical procedures and requirements of the CPG's QAPPs written by Windward, *Caged Bivalve Study, Addendum to the QAPP: Surface Sediment Chemical Analyses and Benthic Invertebrate Toxicity and Bioaccumulation Testing* (CPG's Benthic QAPP Addendum No. 4) dated February 8, 2011 for the Caged Bivalve Study:

- Worksheet No. 1 contains the title and approval pages for the addendum
- Worksheet No. 2 contains the QAPP identifying information
- Worksheet No. 3 provides the distribution list
- Worksheet No. 10 describes the specific problem definition
- Worksheet No. 11 provides the project quality objectives
- Worksheet No. 14 provides a summary of project tasks
- Worksheet No. 16 provides the schedule and timeline
- Worksheet No. 18 provides the proposed sampling locations
- Worksheet No. 37 provides the usability assessment (field summary report)

Worksheets 12, 15, 19, 20, 23, 24, 28, 30, and 36 for all analyses are covered in the CDM QAPP Addendum No. 5, Fish Tissue Sampling. For the polyaromatic hydrocarbons (PAH) analysis, worksheets 12, 15, 19, 20, 23, 24, 28, 30, and 36 are updated and included in this addendum to address the revised analytical requirements for this event. The CPG's QAPPs provide procedures for the caged bivalve study.

1.1 Summary of Bivalve Tissue Sample Acceptance

CDM's oversight program is designed to provide technical review, verify the accuracy of the CPG's in situ caged bivalve study and evaluate the CPG-implemented QAPPs for bivalve tissue sampling and analysis.

The CPG is performing the in situ caged bivalve study to determine the potential for these organisms to be used as a long-term tool for monitoring chemicals in the water column prior to and following remediation activities in the LPRSA. In addition, chemical concentrations in bivalve tissue will be used to assess the effects of LPRSA chemicals on bivalves and as a component in a food web model. CDM will accept split samples of bivalve tissue homogenate

from CPG's laboratory, Alpha Analytical. Split samples will be analyzed for select contaminants as requested by EPA and USACE as follows:

- polychlorinated biphenyl (PCB) congeners
- polychlorodibenzodioxin/furan (PCDD/PCDF) congeners
- PAH compounds
- organochlorinated pesticides
- semivolatile organic compounds (SVOCs) including phthalates
- metals (including total and methylmercury)
- percent lipids and percent moisture

Split samples will not be accepted for the following analytes which will be analyzed by the CPG contractors: alkylated PAHs, PCB Aroclors, and butyltins. This oversight FINAL QAPP details the planning and execution processes for accepting, preparing and shipping samples for analysis.

QAPP Worksheet #1
Title and Approval Page

Document Title: **LPR Restoration Project Quality Assurance Project Plan (QAPP) Final Addendum No. 7, Caged Bivalve Study**

Lead Organization: United States Army Corps of Engineers (USACE) – Northwestern Division

Preparer's Name and Organizational Affiliation: Christine Julias, CDM

Preparer's Address, Telephone Number, and E-mail Address: 100 Crossways Park West, Suite 415 Woodbury, NY 11797; (516) 730-3941; JuliasC@cdm.com

Preparation Date (Day/Month/Year): May 2, 2011

Investigative Organization's Project Manager/Date: _____ Signature

Frank Tsang/CDM

Investigative Organization's Project QA Manager/Date: _____ 5-2-11 Signature

Jo Nell Mullins/CDM

Lead Organization's Project Manager/Date: _____ Signature

Elizabeth Buckrucker/USACE – KC District

EPA Remedial Project Manager /Date: _____ Signature

Stephanie Vaughn

EPA Quality Assurance Officer /Date: _____ Signature

William Sy

Document Control Numbering System: Not Applicable (N/A)

QAPP Worksheet #2
QAPP Identifying Information

Site Name/Project Name: Lower Passaic River (LPR) Restoration Project	**Title**: Final QAPP Addendum No. 7, Caged Bivalve Study
Site Location: LPR study area, New Jersey	**Revision Number:** 1
Site Number/Code: NJD 980528996	**Revision Date:** NA
Operable Unit (OU): OU2	**Contractor Name:** CDM
Contractor Number: W912DQ-08-D-0018	
Contract Title: Unrestricted Indefinite Delivery/Indefinite Quantity, Multiple Award Contract, for Achitect-Engineer (AE) Environmental Services for EPA Region 2 and the Corps of Engineers Northwestern Division.	
Task Order Number: 14	

1. Regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) (Superfund)
2. Approval entity: United States Army Corps of Engineers (USACE)
3. The QAPP is (select one): Generic √ **Project Specific**
4. Dates of negotiation: NA
5. Dates and titles of QAPP documents written for previous and current site work, if applicable:

Title	Approval Date
See Final QAPP for a full list of previous QAPP prepared for site work	
Lower Passaic River RI/FS Oversight Final QAPP, Physical Water Column Monitoring and Generic Information for Upcoming Tasks (PWCM/Generic QAPP) (referred to herein as Final QAPP)	March 2010
LPR RI/FS Oversight QAPP, Final Addendum No.1: *Avian Community Survey*	August 6, 2010
LPR RI/FS Oversight QAPP, Final Addendum No.2: *Fish Community Survey*	June 8, 2010
LPR RI/FS Oversight QAPP, Final Addendum No.3: *Benthic Invertebrate Community Survey*	June 8, 2010
LPR RI/FS Oversight QAPP, Addendum No.4: *Surface Sediment Sampling Co-located with the Small Forage Fish Tissue Samples during the Summer 2010 Benthic Community Survey oversight*	July 12, 2010
LPR RI/FS Oversight QAPP, Addendum No.5: *Fish Tissue Analysis*	August 24, 2010
LPR RI/FS Oversight QAPP, Addendum No.6: *Habitat Identification Survey*	August 9, 2010

6. Organizational partners (stakeholders) and connection with lead organization: EPA, USACE, New Jersey Department of Environmental Protection (NJDEP), New Jersey Department of Transportation (NJDOT), National Oceanic Atmospheric Administration (NOAA), United States Fish and Wildlife Service (USFWS)

7. Data users: EPA, USACE, Partner Agencies, CDM, Louis Berger Group, Inc., HydroQual, Inc., and stakeholders

8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below: the Final QAPP and QAPP Addendum No. 5 provides all the required worksheets. *This addendum addresses only the caged bivalve study; therefore, only worksheets pertinent to this task and information not previously provided are included.*

QAPP Worksheet #3
Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address
Stephanie Vaughn	Remedial Project Manager (RPM)	EPA	(212) 637-4427	(212) 637-4393	vaughn.stephanie@epamail.epa.gov
Elizabeth Buckrucker	Project Manager (PM)	USACE	(816) 389-3581		elizabeth.a.buckrucker@usace.army.mil
William Sy	QA Officer	EPA	(732) 632-4766	(732) 321-6622	sy.william@epa.gov
Janine MacGregor	Partner Agency	NJDEP	(609) 633-0784		janine.macgregor@dep.state.nj.us
Elkins Green	Partner Agency	NJDOT	(609) 530-8075		elkins.green@dot.state.nj.us
Tim Kubiak	Partner Agency	USFWS	(609) 646-9310		tim_kubiak@fws.gov
Reyhan Mehran	Partner Agency	NOAA	(212) 637-3257		reyhan.mehran@noaa.gov
Jeanne Litwin	Project Manager	CDM	(212) 377-4524	(212) 785-6114	LitwinJ@cdm.com
Frank Tsang	Project Manager	CDM	(212) 377-4056	(212) 785-6114	TsangC@cdm.com
Sharon Budney	Deputy Project Manager	CDM	(732) 590-4662	(732) 225-7851	BudneySL@cdm.com
Jeniffer Oxford or other assigned QAC	Regional QA Coordinator (RQAC)/ Project QA Officer	CDM	(212) 377-4536	(212) 785-6114	OxfordJM@cdm.com
George Molnar	Field Task Leader	CDM	(732) 590-4633	(732) 225-7851	MolnarGC@cdm.com
Scott Kirchner	Analytical Services Coordinator	CDM	(732) 590-4677	(732) 225-7851	KirchnerSF@cdm.com
James Fitzpatrick	Sediment Transport Modeler	HydroQual	(201) 529-5151	(201) 529-5728	jfitzpatrick@hydroqual.com
Candice Navaroli	Laboratory Manager	Axys Analytical Services Ltd.	(250) 655-5800 or (888) 373-0881	TBD	cnavaroli@axys.com
Lynda Huckestein	Laboratory Manager	Columbia Analytical Services Inc. (CAS).	(360) 507-3358 or (360) 577-7222	TBD	lhuckestein@caslab.com
Nisreen Saikaly	Laboratory Project Manager	CDM Subcontract Laboratory-Shealy	(800) 673-9375 ext 106	(803) 791-9111	NSaikaly@Shealylab.com

QAPP Worksheet #10
Problem Definition

The problem to be addressed by the project: The CPG is conducting this study to determine the potential for using caged bivalves as a long-term tool for monitoring chemicals in the water column prior to and following remediation in the LPRSA. In addition, chemical concentrations in caged bivalves tissue will be used to assess the effects of LPRSA chemicals on bivalves and as a component in a food web model. CDM oversight and analysis will provide verification of CPG compliance with their approved project plans and accuracy of the derived data.

Oversight will include:

- Acceptance of split tissue homogenate from CPG's laboratory, Alpha Analytical
- Review of CPG-selected sampling locations
- Review of bivalve measurements, chemical data and condition of surviving organisms from each sampling location during scheduled cage checks and at study termination, as applicable
- Review of health assessments conducted on sacrificed individuals representative of tests species used prior to deployment, during scheduled cage checks and at study termination
- Coordination with the CPG-designated laboratory, Alpha Analytical, to collect government split samples of tissue (refer to Worksheet 11 for details on split samples and refer to Worksheet 19 for minimum mass requirements)

The environmental questions being asked:

- Does the CPG data adequately describe the site conditions and is it representative for project decisions?
- Are the CPG and CDM data complete and accurate?
- Are the data sets comparable?
- Are the relative percent differences (RPDs) between the CPG and CDM data within the measurement performance criteria?

Secondary data: See Worksheet 13 of the CPG Benthic QAPP (Windward 2009)

The possible classes of contaminants and the affected matrices:

Split tissue samples will be collected for the following chemical analyses:

- PCB congeners
- PCDD/PCDF congeners
- chlorinated pesticides
- PAHs, and TCL SVOCs
- metals including total and methylmercury
- percent lipids and percent moisture

QAPP Worksheet #10
Problem Definition

Split samples will not be accepted for the following analytes which will be analyzed by the CPG contractors: alkylated PAHs, PCB Aroclors, and butyltins.

The rationale for inclusion of chemical and non-chemical analyses:

The split samples will be used to support the goals of the oversight program. The split sample analyses were determined to be more critical for oversight evaluation; the analyses that will not be split are ancillary parameters and not major risk drivers. The field observations and split sample data will enable CDM to perform technical review and evaluation on the CPG field program, analytical data and reports and to assess any potential bias in the CPG dataset.

Project decision conditions ("If..., then..." statements):

- If sample results are not comparable with the CPGs, then CDM will note deviations in the Data Reports submitted to USACE and EPA. The CDM Project Manager, USACE PM and EPA RPM will be informed if there are deviations.
- If the CPG team needs to reprioritize analytical parameters, change compositing procedures, or if there are any changes to the planned analytical program, CDM will communicate this change to the USACE and EPA and document it in the Data Reports.

CDM will present the data findings in a Data Report and submit it to the USACE and EPA who will then determine if any additional actions are required.

QAPP Worksheet #11
Project Quality Objectives /Systematic Planning Process Statements

Who Will Use the Data? USACE, EPA and other partner agencies, CDM, and stakeholders (as necessary).

What Will the Data be Used For?

The CPG will use the study to evaluate the potential for caged bivalves to be used as a long-term monitoring tool of chemicals in the water column of the LPRSA. In addition, chemical concentrations in tissue will be used to assess the effects of LPRSA chemicals on bivalves and as a component in a food web model. Oversight activities will monitor the CPG-implemented caged bivalve study, tissue sampling, and analytical program to verify that elements of the approved RI/FS QAPPs are fulfilled. The CDM field crew will also review the CPG-selected sampling locations and compositing procedures. CDM's split sample results will be compared to the data obtained by the CPG to determine if a bias exists in the data produced by the CPG and if the data is complete, accurate and compliant with the approved QAPPs.

A comparison of the split sample data and the CPG parent sample data will only be completed for parameters that were analyzed and detected by both the CPG program and the oversight program. Data comparison will not be conducted on concentrations that are non-detect by the oversight data validators. (Note that if a consistent bias in detections is observed in either the split samples or CPG samples, an evaluation of detection limits will be completed.) The data comparison will be presented in a table showing the relative percent difference for values that are 5 times the quantitation limits. As appropriate, alternative data comparisons will be provided. For each location, a mean and variance of the sample concentrations may also be calculated. These statistics will be compared to the CPG samples. For analytical groups that contain multiple parameters (e.g., congeners), the data comparison will be completed on select parameters per chemical class. Parameters will be selected by the project chemist/and analytical service coordinator to cover a range of concentrations from non-detects to high concentrations. In addition analytes of greater risk or of greater concern will be selected for comparison over other analytes. This selection will be made with the consensus of the USACE and EPA.

Because of the overlap of the SVOC and PAH chemical classes, some analytes will be reported twice in the split sample program. For the data comparison, PAH-SIM results reported by CAS Laboratory (CDM's subcontract laboratory) will take precedence over the PAH data generated by DESA/ EPA CLP or CDM Subcontract laboratory during the SVOC analysis.

CDM's QC data will be used to determine CDM's split sample data quality and comparability with the CPG's data and whether sample results are acceptable based on the established project data quality objectives (DQOs). QC sample results will be compared to the measurement performance criteria (MPC) of the data quality indicators (DQIs).

To further achieve these objectives, CDM field personnel will observe and monitor the CPG contractor's implementation of the RI/FS QAPPs and will note any deviations. Deviations will be brought to the attention of the CPG's contractor, and reported to the CDM project manager who will communicate this information to the USACE PM and EPA RPM. These will be documented in ongoing and Final Reports and include a discussion of the impact of the deviation(s) on the data quality. The CPG contractor's activities will be documented in the field logbook and oversight forms. A copy of the oversight form is provided in Appendix B of CDM's Final QAPP.

What Type of Data is Needed?

CDM may observe and document the caged bivalve and tissue compositing activities conducted by the CPG's contractor. Split samples will be collected at locations for which sufficient tissue mass is available, by mutual agreement of CDM and the CPG contractor or as directed by the CDM Deputy PM or the USACE/EPA project managers.

Chemical data, PCB congeners, PCDD/PCDF congeners, organochlorine pesticides, PAHs, SVOCs, metals (including total mercury, and methylmercury), percent lipids and percent moisture, will be determined from the split samples accepted from the CPG. Low limits are required for mercury and methylmercury as shown on QAPP worksheet No. 15.

QAPP Worksheet #11
Project Quality Objectives /Systematic Planning Process Statements

How much data are needed?

CDM will accept split samples at approximately 10 percent of the sampling locations. Worksheets No. 11 and 18 of the CPG's Benthic QAPP Addendum No. 4 and Figure 1 show the planned locations for sampling.

Approximately 10 percent of the samples will be split to determine if a bias exists in the data produced by the CPG. Oversight activities are listed in Worksheet 10. The split sample program includes tissue split samples from the CPG laboratory. Field duplicates will be analyzed if sufficient sample mass is available.

How "good" do the data need to be in order to support the environmental decision?

Definitive level data is required to produce the data quality required for risk assessments, full validation of the data and to enable comparison with the CPG generated data set. Fixed based laboratories with EPA, Environmental Laboratory Accreditation Program (ELAP) or national ELAP (NELAP) certifications and qualification will be used to generate the analytical data. CDM's oversight staff will document whether the in situ caged bivalve study is in compliance with the CPG's Benthic QAPP Addendum No. 4. The representativeness of the data is dependent on the sampling design established by the CPG. Split samples will consist of a portion of tissue homogenate, of sufficient volume to fulfill analytical needs.

The laboratory reporting limits (contract required quantitation limits (CRQLs)) for CLP data, or reporting limits for subcontract laboratory data), need to be below or equal to the CPG's project required quantitation limits goals or the CPG's achievable laboratory quantitation limits. CDM will notify EPA's RSCC or the subcontract laboratory and request lower reporting limits to achieve the project data quality objectives for sensitivity as needed.

Validation of data will be performed by DESA/ EPA; however, samples analyzed by a subcontract laboratory will be validated by CDM.

In addition, to ensure that measurement performance criteria for usability (criteria for DQIs) are met, all CDM data will be subject to a data usability assessment. The inputs will be the EPA generated validation reports and subcontract laboratory QC summaries. Measurement performance criteria presented in Worksheets No.12, 28, 35 and 36 will be evaluated as discussed on Worksheet No.37. The results will be presented in a CDM data report.

The data usability assessment will evaluate whether appropriate field procedures were followed and whether data met the approved QAPP and project DQOs, and are usable for the stated project needs.

Where, when, and how should the data be collected?

When – Split tissue samples will be accepted from the CPG's contractor laboratory. The CPG laboratory will ship the split tissue samples to CDM's laboratories after the late spring 2011 tissue collection event and after the samples have been processed and composited for analysis. This will be performed according to the CPG's schedule. The exact sample processing dates are to be determined.

Where – The bivalves will be collected from the LPRSA locations shown on Figure 1. Tissue processing will be performed at the CPG's laboratory, Alpha Analytical. Samples will be split where sufficient tissue mass was generated for both sample sets.

How – Tissue processing procedures are described in the CPG's Benthic QAPP (Windward 2009) (various worksheets) and CPG's Benthic QAPP Addendum No. 4

QAPP Worksheet #11
Project Quality Objectives /Systematic Planning Process Statements

and its Attachment U.

Who will collect and generate the data?

CDM oversight staff will record field observations in logbooks. The CPG's laboratory, Alpha Analytical will split a portion of homogenate for split samples and ship to the appropriate laboratory. The analytical laboratories outlined in this Final QAPP Addendum will generate the data.

How will the data be reported?

- Accepted tissue composite samples will be recorded as described in CDM's Final QAPP using field logbooks in accordance with TSOP 4-1 provided in Appendix C of the Final QAPP.
- Results will be reported in text format and will include a discussion of the data quality, deviations from the QAPP, and oversight data comparability with the CPGs data. This review will be used to evaluate the accuracy of the CPG data.
- To ensure comparability of the data sets, CDM will obtain lipid results generated by the CPG's analytical laboratory using the Bligh Dyer method and use them to generate lipid corrected dioxin and PCB Congener data in the event that the lipid results generated by CDM's subcontract laboratory differ from the CPG's data. If necessary, both sets of data will be reported, results calculated with CDM lipid results and those calculated with the CPG lipid results.
- Sample results generated by the DESA or EPA CLP laboratory will be e-mailed to CDM for use in the data assessment and evaluation
- Sample results generated by CDM's subcontract laboratory will be e-mailed to CDM for review and validation.
- Data reporting is further covered in the Final QAPP.

How will the data be archived?

- Hard copies of data will be kept in the CDM Edison office until archived in the project file; if requested, survey data will be uploaded to a PREmis or equivalent database.
- The Final QAPP contains other archival information.

QAPP Worksheet #12
Measurement Performance Criteria Table [2]

Matrix	Tissue
Analytical Group	PAHs (SVOC-SIM)
Concentration Level	Low

Sampling Procedure	Analytical Method/ SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria[1] (MPC)	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
CPG Group's SOP, and QAPP CDM will accept split samples	CAS SOP for PAH analysis: SOC-8270P, Rev. 7 See Appendix L	Precision	RPD ≤ 40% if concentration ≥5 CRQL	Split samples and field duplicates	S & A
		Precision	Per laboratory SOP (Attachment A of Appendix L)	Laboratory duplicate or MS/MSD	A
		Accuracy/Bias	Various %recoveries (See Attachment A of laboratory SOP for individual limits)	Matrix Spike	S & A
		Accuracy/Bias		Laboratory Control Sample	A
		Accuracy/ Representativeness	4±2 degrees Celsius 10 degrees Celsius (DV)	Temperature Blank checks Data validation (DV)	S
		Comparability	Comparable units, and methods	Evaluated during DQA	S & A
		Completeness	≥ 90% Collection and ≥ 90% Valid data	Evaluated during DQA	S & A
		Sensitivity/ Accuracy	≤ QLs (WS No.15) and Table 4 of laboratory SOP)	Field rinsate/ Method blanks assessed during DV and DQA	S & A

Note:
1. The laboratory must perform and meet all the quality assurance requirements specified in the laboratory method SOP.
*Surrogates are pure analytes added to every blank, sample, matrix spike, matrix spike duplicate, and standard in known amounts before extraction or other processing; used to evaluate analytical efficiency by measuring recovery.
2. All other worksheets are included in the QAPP Addendum No. 5.

QAPP Worksheet #14
Summary of Project Tasks

Sampling Tasks:
The CPG's contractor laboratory will ship split samples of homogenized bivalve tissue to CDM's subcontract laboratories. On behalf of the USACE and EPA, the oversight program is designed to provide technical review and evaluation of CPG-implemented field plans. Worksheet 10 discusses the oversight activities for the sampling activity, and Worksheet 11 provides details on the data to be collected. CDM will observe and document the activities conducted during the study. CDM will accept split tissue samples as volume requirements allow.

Analysis Tasks:
Split samples will be collected from homogenized tissue composites to be generated by the CPG's subcontract laboratory, Alpha Analytical.

Analyses on tissue samples will include PCB congeners, PCDD/PCDF congeners, chlorinated pesticides, PAH compounds, SVOC compounds, metals (total and methylmercury), percent lipids, and percent moisture.

Quality Control Tasks: CDM will observe CPG's processing and handling of the tissue samples. CDM will accept splits and one field rinsate blank of the blender used to homogenize the samples. The CDM Deputy Project Manager or designee will review the logs to ensure that the required information has been documented.

Secondary Data: Since this is an oversight project, no secondary data is being used directly by CDM. Data generated by the CPG - field program will be used as shown on worksheet 11 of the CPG's Benthic QAPP Addendum No. 4: Caged Bivalve Study QAPP.

Data Management Tasks:
Analytical data generated by the various laboratories will be managed according to the procedures described in the Final QAPP.

Documentation and Records: Records of accepted tissue samples will be documented in accordance with TSOP 4-1 provided in Appendix C of the Final QAPP. The Tissue Analysis results will be documented in the following:

1. Data Validation reports
2. COCs, ANSETS, and Trip Report
3. Oversight summary report
4. Data Quality and Usability Summary Report

Assessment/Audit Tasks: See Final QAPP for assessment tasks (CDM 2009)

Data Review Tasks: The CPG's Data Summary Repot will be reviewed by CDM. A data quality evaluation will be performed based on the CPG's compliance with the approved QAPP. A comparison of CDM's and the CPG's tissue sample results will be included in the data quality evaluation and submitted to the USACE.

QAPP Worksheet #15
Reference Limits and Evaluation Table

Matrix: Tissue
Analytical Group: PAH by CAS SOP for PAH analysis: SOC-8270P, Rev. 7
Concentration Level: Low (µg/kg)

Analyte	CAS Number	Project Action Limit[1]	Project Quantitation Limit Goal[2]	Analytical Method[3]			Achievable Laboratory Limits[3]	
				MDLs	SOM01.2 QL	8270 QL	MDLs	QLs
1-Methylnaphthalene	90-12-0	TBD	937000	NA	Not Listed	Not Listed	0.30	0.5
1-Methylphenanthrene	832-69-9	TBD	Not Available	NA	Not Listed	Not Listed	0.082	0.5
2,3,5-Trimethylnaphthalene	2245-38-7	TBD	Not Available	NA	Not Listed	Not Listed	0.24	0.5
2,6-Dimethylnaphthalene	581-42-0	TBD	Not Available	NA	Not Listed	Not Listed	0.41	0.5
2-Methylnaphthalene	91-57-6	TBD	337000	NA	170	660	0.44	1.0
Acenaphthene	83-32-9	TBD	240	NA	170	660	0.11	0.5
Acenaphthylene	208-96-8	TBD	240	NA	170	660	0.069	0.5
Anthracene	120-12-7	TBD	240	NA	170	660	0.065	0.5
Benzo[a]anthracene	56-55-3	TBD	240	NA	170	660	0.066	0.5
Benzo[a]pyrene	50-32-8	TBD	240	NA	170	660	0.081	0.5
Benzo[b]fluoranthene	205-99-2	TBD	240	NA	170	660	0.070	0.5
Benzo[e]pyrene	192-97-2	TBD	Not Available	NA	Not Listed	Not Listed	0.062	0.5
Benzo[g,h,i]perylene	191-24-2	TBD	240	NA	170	660	0.073	0.5
Benzo[j]fluoranthene [4]	205-82-3	TBD	Not Listed	NA	Not Listed	Not Listed	0.1	0.5
Benzo[k]fluoranthene	207-08-9	TBD	240	NA	170	660	0.056	0.5
Chrysene	218-01-9	TBD	240	NA	170	660	0.076	0.5
Dibenzo[a,h]anthracene	53-70-3	TBD	240	NA	170	660	0.059	0.5
Dibenzothiophene [5]	135-65-0	TBD	293000	NA	Not Listed	Not Listed	0.23	1.0
Fluoranthene	206-44-0	TBD	240	NA	170	660	0.090	0.5
Fluorene	86-73-7	TBD	240	NA	170	660	0.15	0.5
Indeno[1,2,3-c,d]-pyrene	193-39-5	TBD	240	NA	170	660	0.064	0.5
Naphthalene	91-20-3	TBD	240	NA	170	660	0.40	1.0
Perylene	198-55-0	TBD	Not Available	NA	Not Listed	Not listed	0.064	0.5
Phenanthrene	85-01-8	TBD	240	NA	170	660	0.36	0.5
Pyrene	129-00-0	TBD	240	NA	170	660	0.098	0.5

Notes:

1. At this time, project-specific action levels have not been developed. The CPG used preliminary screening levels to derive Data Quality Levels (DQLs).

2. The PQLGs shown are the DQLs taken from the CPG RI/FS QAPP, *Surface Sediment Chemical Analyses and Benthic Invertebrate Toxicity and Bioaccumulation Testing*, QAPP Addendum Number 4, *Caged Bivalve Study,* February 2011. The split sample data should be low enough for data comparison. Differences in laboratory detection limits will be considered when comparing the data. Actual QLs will differ since the laboratory reports to sample specific detection limits.

3. Achievable MDLs listed are the statistically-derived MDLs. The QLs listed are based on CAS Laboratory's typical sample specific detection limits. Actual QLs may be higher and are dependent on the sample matrix effects. MDLs and QLs are limits that an individual laboratory can achieve when performing the analytical method.

4. This compound may co-elute with benzo(k)fluoranthene.

5. This compound is not listed in SOC 8270P, therefore the related MDL and RL are estimated.

QAPP Worksheet #16
Project Schedule Timeline Table

Activities	Organization	Anticipated Date(s) of Initiation	Anticipated Date of Completion	Deliverable	Deliverable Due Date
Prepare and submit: Oversight QAPP Addendum for Caged Bivalve Study to EPA and USACE	CDM	February 15, 2011	February 23, 2011	UFP-QAPP addendum, Draft	February 24, 2011
Prepare and submit: Final oversight QAPP Addendum for Caged Bivalve Study	CDM	As soon as comments are received	April 28, 2011	UFP-QAPP addendum, Final	May 2, 2011
Acceptance of splits and sample handling activities	CDM	Mid to end of June 2011 – TBD	10 days after commencement date	Summary report of chemical data	To be determined
Laboratory Analysis	CDM subcontract laboratory(ies)	July 2011	To be determined; data collection will be dependent on the CPG schedule	Data Package	To be determined; will be dependent on the CPG schedule For standard analyses, 21 days after the last sample is received; however, specialized analyses may take additional time
Validation and verification of sample data	CDM	August 2011	August 2011	Validated data report	To be determined; will be dependent on CPG schedule
Oversight /Data Evaluation	CDM	To be determined	To be determined	Oversight data Comparison and Summary Report/ Data Quality Summary Report	To be determined
Review Tissue Chemical Analysis Data Report	CDM	90 days after each sampling event	1 month after receipt of report	Comments on Fish Tissue Chemical Analysis Data Report	1 month after receipt of report

QAPP Worksheet #18
Sampling Locations and Methods/SOP Requirements Table

Survey Location ID	Depth	Analytical Group	Concentration Level	Estimated Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
Refer to QAPP prepared by Windward for the CPG	Tissue split samples	Analytical group for split samples includes: PCB congeners, PCDD/PCDF congeners, chlorinated pesticides, PAH and SVOC, metals (including total and methylmercury), and percent lipid	Low	Approximately 10 percent of CPG samples will be split.	Attachment U of Benthic QAPP Addendum No. 4) (Windward 2011) (also see footnotes)	Split samples will be accepted judgmentally by the on-site oversight staff in consultation with the PM and USACE/EPA Selection may be restricted by the species collected during sampling and the amount of tissue mass available.
See CPG's QAPP Worksheet No.18 for the sampling locations and sampling rationale. In order to obtain sufficient mass for analysis of all the required chemical parameters, CDM may split tissue mass across different samples to complete each analytical suite.						

Notes:
Refer to the QAPP prepared by Windward for the CPG (Worksheets No. 10, 11 and 18 and Figure 1) titled*, Surface Sediment Chemical Analyses and Benthic Invertebrate Toxicity and Bioaccumulation Testing*, *Benthic QAPP Addendum No. 4: Caged Bivalve Study* (February 8, 2011) for sampling information.

QAPP Worksheet #19 – Tissue Analysis
Analytical SOP Requirements Table

Matrix[1]	Analytical Group	Concen -tration Level	Analytical and Preparation Method/SOP Reference[2]	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light)[3]	Maximum Holding Time (preparation/ analysis)
CAS Laboratory							
Tissue	Pesticides	Low	EPA 1613B Modified for HRGC/ HRMS (Axys SOP MLA-028, Rev. 5)	Minimum = 30 g (combine mass for pesticide, PCB, PCDD/ PCDF, and PAH) Lipids extracted with organics – no additional mass required	1- 4 oz amber glass jar (ship one jar for pesticide, PCB, PCDD/PCDF, and PAH)	Freeze sample: 0^0C to -20 ^{0}C	14 days to extraction, 40 days to analysis For this study samples can be stored 299 days if frozen; 40 days to extraction.
Tissue	PCDD/PCDF Congeners	Low	EPA 1613B for HRGC/ HRMS (Axys SOP MLA-017)			Freeze sample: 0^0C to -20 ^{0}C	1 year for solid multiphase samples - If stored at less than -10^{0}C. 1 year for sample extracts – if stored at less than -10^{0}C.
Tissue	Percent Lipids	TBD	Axys SOP SLA-020, Rev. 2/ SM 2540B equivalent				
Tissue	PAH	Low	EPA Method 8270C Modified (CAS SOP SOC-8270P Rev.7)			Freeze sample: 0^0C to -20 ^{0}C	14 days to extraction, 40 days to analysis For this study, samples can be stored 199 days if frozen; 40 days to extraction.
Tissue	Percent Moisture	TBD	Axys SOP SLA-015, Rev. 6 / SM2540G Modified	Minimum mass = 10 g	2- 2 oz glass jar		Solid multiphase samples - 1 year If stored at less than -10^0 C
Cape Fear and Microbac Laboratory							
Tissue	PCB Congeners	Low	CF-OA-E-003, Rev.1 based on EPA 1668B for HRGC/ HRMS (Cape Fear SOP)	Minimum mass = 10 g Lipids extracted with organics – no additional mass required	1- 4 oz amber glass jar	Freeze sample: 0^0C to -20 ^{0}C	1 year for solid multiphase samples - If stored at less than -10^{0}C. 1 year for sample extracts – if stored at less than -10^{0}C
Tissue	Percent Lipids	TBD	SOP CF-OA-E-001, Rev. 2.1/ SM 2540B equivalent				
Tissue	TCL SVOC	Low	Microbac SOP 625-8270, Rev. 10/ SW-846 Method 8270C equivalent	Minimum mass = 10 g	1- 8 oz glass jar (ship one jar for SVOC, and metals)	Freeze sample: 0^0C to -20 ^{0}C	14 days to extraction; 40 days to analysis at 4 ^{C}C

QAPP Worksheet #19 – Tissue Analysis
Analytical SOP Requirements Table

Matrix[1]	Analytical Group	Concen -tration Level	Analytical and Preparation Method/SOP Reference[2]	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light)[3]	Maximum Holding Time (preparation/ analysis)
Tissue	Metals (ICP-AES and MS)	Low	Microbac SOP 2007-6010, SW-846 Method 6010B equivalent Microbac SOP 2008-6020 / SW-846 Method 6020 equivalent	Minimum mass = 10 g	Contained in SVOC bottle	Freeze sample: 0^0C to -20 ^{0}C	6 months
Tissue	Total and methyl mercury	Low	Microbac SOP Hg-1631 Rev. 2/ EPA 1631 equivalent and SOP Methyl Mercury Draft, Rev. 0	Minimum mass = 10 g	1- 4 oz pre-tared polyethylene bottle	Cool to 4 ^{C}C ± 2 ^{C}C and freeze as soon as possible [maintain in the dark]	1 year [if aliquoted, weighed and frozen at <-15 ^{C}C]

Notes:

1: Tissue matrix refers to split tissue sample analyzed for chemical concentration.

2: The CDM analytical subcontract laboratory SOPs for these analyses are shown in Appendix K of the Oversight QAPP, Addendum No. 5: Fish Tissue Analysis and in Appendix L of this Final QAPP. The Axys laboratory SOPs are proprietary but SOP summaries are included in the Oversight QAPP, Addendum No.5.

3. The actual jar size may vary depending on the need of the assigned laboratory. The sampler should confirm sample volumes with the laboratory prior to mobilizing to the field.

QAPP Worksheet #20
Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concen-tration Level	Analytical and Preparation SOP Reference	No. of Split Sampling Locations	No. of Field Duplicate Pairs	No. of Extra Volume Laboratory QC (e.g., MS/MSD) Samples	No. of Equipment Rinsate Blanks	No. of Trip. Blanks	No of PE Samples	Total No. of Samples
Tissue	PCB congeners	Low	EPA Method 1668B for HRGC/HRMS /Cape Fear SOP CG-OA-E-003	Sample locations and number of samples to be determined (TBD)	1 per 20 or less (total of 1)	0	1	0	0	10% Splits, exact numbers TBD
Tissue	PCDD/PCDF congeners	Low	EPA Method 1613B – Axys SOP MLA-017	TBD	1 per 20 or less (total of 1)	0	1	0	0	TBD
Tissue	Chlorinated Pesticides	Low	EPA Method 1613B Modified by HRGC/ HRMS: Axys SOP MLA-028	TBD	1 per 20 or less (total of 1)	1 per 20 or less (total of 1)	1	0	0	TBD
Tissue	PAHs	Low	CAS SOC-8270P	TBD	1 per 20 or less (total of 1)	1 per 20 or less (total of 1)	0	0	0	TBD
Tissue	TCL SVOCs	Low	Microbac SOP for SW-846 Method 8270C	TBD	1 per 20 or less (total of 1)	1 per 20 or less (total of 1)	1	0	0	TBD
Tissue	TAL metals	Low	Microbac SOP for SW-846 Method 6010B/ and 6020	TBD	1 per 20 or less (total of 1)	1 per 20 or less (total of 1)	1	0	0	TBD
Tissue	Mercury and methylmercury	Low	Microbac SOP for EPA Method 1630/ and 1631	TBD	1 per 20 or less (total of 1)	1 per 20 or less (total of 1)	1	0	0	TBD
Tissue	Percent Lipid/ Percent moisture	Low	AXYS Modified SM2540B/ SM2540G	TBD	1 per 20 or less (total of 1)	1 per 20 or less (total of 1)	1	0	0	TBD

Notes:

1. The FASTAC decision process is required for obtaining laboratory services but tissue sample analysis is not available via EPA. Chlorinated pesticides, dioxin/furans, and moisture will be analyzed by Axys laboratory. The Axys subcontract laboratory will be used due to the difficulty of analyzing the sample matrix for the selected analyses in order to ensure accurate results, to reduce uncertainties in the measurements and to obtain data comparable with data from previous and future surveys. Another subcontract laboratory is being procured to perform the remaining tissue analysis. CDM subcontracted one of its master services agreement laboratories, Shealy, to obtain analytical services for the remaining analyses. PAHs will be analyzed by Columbia Analytical Services.
2. The exact number of samples to be split will be determined by the CPG bivalve tissue collection during the late summer 2011 effort.

QAPP Worksheet #23
Analytical SOP References Table

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
CAS SOP: SOC-8270P	*Polycyclic Aromatic Hydrocarbons (PAH) by GC/MS Selective Ion Monitoring, EPA 8270C SIM.* February 2008. (Reviewed annually; last reviewed April 2010)	Definitive	PAH-SIM	GCRMS	Columbia Analytical Services, Inc.	No

Notes:

As necessary, the assigned laboratories will perform additional clean-up of split samples (via gel permeation chromatography) prior to analysis of organic compounds.

QAPP Worksheet #24
Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/ MS	Initial Calibration and calibration verification check: Per laboratory SOP	After set up, prior to run and after instrument changes or failures of checks.	15 % RSD and not less than 30% for any compound; ± 20% recovery per laboratory SOP.	Check, correct; re-calibrate and rerun all samples analyzed after last valid calibration check	Laboratory analyst / QA officer - TBD	CAS SOP for PAH analysis: SOC-8270P, Rev. 7
	Calibration checks: continuing calibration verifications (CCVs) per laboratory SOP	Daily: Beginning of run and after every 10 samples and at end of analytical run	± 20% recovery per laboratory SOP.	Check, correct; re-calibrate and rerun all samples analyzed after last valid calibration check	Laboratory analyst / QA officer - TBD	

Notes:

1. General GC/MS calibration requirements are presented. Instruments used for analyses follow the calibration frequencies outlined in the method SOPs (Appendix K of the Oversight QAPP, Addendum No. 5) and Appendix L of this Final QAPP Addendum No. 7. Laboratory specific calibration information is maintained by the laboratories; method specific calibration information is detailed in the methods.

QAPP Worksheet #28
QC Samples Table for Fish Tissue Sampling

Matrix	Tissue
Analytical Group	PAH
Concentration Level	Low
Sampling SOP(s)	See worksheet No.21 – split of CPG samples
Analytical Method/SOP Reference	CAS Laboratory SOP for PAH analysis: SOC-8270P, Rev. 7 (SVOC – SIM)
Sampler's Name	TBD
Field Sampling Organization	CDM
Analytical Organization	Columbia Analytical Services
No. of Sample Locations	See worksheet No.18 & 20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	per extract batch	Per laboratory SOP	Investigate and correct per laboratory SOP	Laboratory Analyst	Accuracy/Sensitivity	No analyte > QL. No target analytes ≥1/2 Method Reporting Limit
Laboratory Duplicate (MS/MSD)	1 per 20 samples	Per laboratory SOP	Investigate and correct; reanalyze affected samples. Flag outliers	Laboratory Analyst	Precision	40% RPD
Matrix Spike	1 per 20 samples or with each group of field samples	Per laboratory SOP	Investigate and correct. Document in data summary	Laboratory Analyst	Accuracy/Precision	Recovery per laboratory SOP (Attachment A of Appendix L)
Laboratory control samples / Surrogate	Every field and QC sample, standards, blanks	Per laboratory SOP	Identify source of problem, make other adjustments and reanalyze	Laboratory Analyst	Accuracy	Recovery per laboratory SOP (Attachment A of Appendix L)
Sample splits and field duplicates	1 per 20 samples	None	Data assessor to inform PM if MPC is exceeded; address in data quality assessment	CDM ASC	Precision	≤ 40% RPD (for results ≥ 5*QL)
Temperature Blank	1 per cooler	≤ 6 degrees Celsius	Note outlier in laboratory narrative. Inform CDM of failure and need for additional coolant; check packing procedure	Laboratory Analyst	Accuracy/bias	≤ 10 degrees Celsius for data validation

All other analyses are included in Worksheet No.28 of QAPP Addendum No.5

Worksheet #30
Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Location/ ID Numbers	Analytical SOP	Validated Data Package Turnaround Time Laboratory/Data Validation	Laboratory/ Organization (Name, Address, Contact person and Telephone Number) [1]	Backup Laboratory/Organization (Name and Address, Contact person and Telephone Number
Tissue	PCB Congeners	Low	TBD	EPA Method 1668B	65 days (35 days /30 days)[2]	Cape Fear (lower tier to Shealy)	EPA Headquarters laboratory
Tissue	PCDD/PCDF	Low		EPA Method 1613B – Axys SOP MLA-017	65 days (35 days /30 days)[2]	Axys Analytical Services Ltd.	Cape Fear
Tissue	Chlorinated Pesticides	Low		EPA Method 1613B modified for HRGG/HRMS (Axys SOP MLA-028)	65 days (35 days for laboratory analysis/ 30 days for data validation)[2]	Axys Analytical Services Ltd.	None
Tissue	PAH	Low		CAS SOP SOC-8270P	65 days (35 days /30 days)[2]	Columbia Analytical Services Inc. 1317 South 13th Avenue, Kelso, Washington 98626	None
Tissue	Percent Moisture	Medium		SM2540G/ Axys SOP EGN007-07	see above	Axys Analytical Services Ltd.	None
Tissue	Percent Lipids	Low		SM2540B Modified	51 days (21 days / 30 days)	Axys Analytical Services Ltd.	None
Tissue	TCL SVOCs	Low		SOM01.2 or SW846 Method 8270C	51 days (21 days / 30 days)	Microbac (lower tier to Shealy)	None
Tissue	Metals	Low		SW846 Method 6010B/6020	51 days (21 days / 30 days)	Microbac (lower tier to Shealy)	None
Tissue	Total Mercury /Methyl mercury	Low		EPA Method 1630/1631	51 days (21 days / 30 days)	Microbac (lower tier to Shealy)	None

Notes:
1. Subcontract laboratories will communicate with the ASC on split sample status and potential analytical difficulties (if any arise).
2. With the approval of the ASC and Deputy PM, the turn-around-time for the laboratory data package deliverable can be adjusted to account for re-analysis or additional quality control as necessary.

QAPP Worksheet #36
Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria[1, 3]	Data Validator (title and organizational affiliation)
IIa /IIb	Tissue	Chlorinated Pesticides –EPA 1613B Modified	Low-trace	Region 2 - National Functional Guidelines[2]	CDM
IIa /IIb		PCB Congeners –EPA 1668B	Low	Region 2 - Data Validation Guidelines SOP HW-46, rev 0 or National Functional Guidelines[2]	EPA Region 2
IIa /IIb		PCDD/PCDF Congeners – EPA 1613B	Low	EPA SOP HW-19 or 25, Validating PCDD/PCDF by HRGC/HRMS, Revision 1 or National Functional Guidelines[2]	CDM ASC, Scott Kirchner or designee
IIa /IIb		PAH – CAS Laboratory SOP	Low-trace	National Functional Guidelines[2]	CDM ASC, Scott Kirchner or designee
IIa /IIb		TCL SVOCs - 8270C	Low	Region 2 – Data Validation Guidelines SOP HW-35, rev 1 or National Functional Guidelines[2]	DESA or ESAT
IIa /IIb		Metals - 6010B/6020	Low/Medium	Region 2 - Data Validation Guidelines SOP HW-2, rev 13 or National Functional Guidelines[2] either will be modified by QAPP worksheets 12,15,19 and 24	DESA or ESAT
IIa /IIb		Methyl Mercury - EPA 1630	Trace		CDM ASC, Scott Kirchner or designee
IIa /IIb		Total Mercury - EPA 1631	Trace		CDM ASC, Scott Kirchner or designee

Notes:

1. Results will be validated if analyzed by a subcontract laboratory by the process of data verification and assessment utilizing the laboratory QC summaries.
2. All validation procedures will utilize the measurement performance criteria in the QAPPs and any additional method requirements.
3. Moisture and percent lipids will not require validation.

QAPP Worksheet #37
Usability Assessment

An Oversight Summary Report and Data Quality Summary Report will be prepared by CDM personnel. Frank Tsang, Project Manager, will be responsible for its content and for assigning this task to CDM personnel. The data comparability review and usability assessment will be conducted on validated data. The effectiveness of control actions will be evaluated during the laboratory review of the data, data validation and data evaluation and data quality assessment process. Data information will be documented in the laboratory narrative, data validation report and in the Data Comparability Report. The report will include an overall assessment of the CPG's analytical data using the results of the split sampling and field oversight including the field oversight observations of deficiencies and compliance; and an assessment of the split sampling data quality. The following items will be assessed for CDM split samples and conclusions drawn based on their results:

Precision – Results of laboratory duplicates will be assessed during data validation and data will be qualified according to the data validation procedures cited on Worksheet No.36. Split samples will be compared by matrix using the relative percent difference (RPD) for each pair of results reported above quantitation limits (QL) or for organic and inorganic analyses respectively. RPD acceptance criteria of less than or equal those listed in this Final QAPP will be used to access sampling precision. Absolute difference will be used when one or both results are at or below the QL. An absolute difference of less than five times the QL will be the acceptance criteria. A discussion summarizing the results of laboratory precision and any limitations on the use of the data will be described in the report.

Accuracy/Bias Contamination – Results for all laboratory blanks will be assessed as part of the data validation. During the validation process, the validator will qualify the data following the procedures described on Worksheet No.36. A discussion summarizing the results of laboratory accuracy and bias based on contamination will be presented and any limitations on the use of the data will be described in the report.

Representativeness –The representativeness of the survey data will be evaluated based on the ability to implement the fish tissue sampling as written in the QAPP. A determination will be made based on the observations completed during the surveys, whether the data results accurately represent the fish tissue concentrations in the study area, and whether the results are comparable with those made in previous events.

Comparability –The results of this oversight will be used in conjunction with the CPG's data to support the investigation results. The data will be handled, analyzed and reported in a manner that is comparable to the CPG's data set. The RPD between CDM's and the CPG's data will be calculated.

Completeness – A completeness check will be performed on the split sample data generated by the laboratories. Completeness will be determined based on whether all CPG planned (or modified) sampling locations were sampled at the pre-determined frequencies and the obtained data set compared to the project completeness goal of 90 percent. A discussion summarizing the results of project completeness and any limitations on the use of the data will be described in the report.

For sampling, completeness will be calculated as the number of samples collected and analyzed divided by the number of samples planned for collection. For each analyte, completeness will also be calculated as the number of data points that meet measurement performance criteria divided by the total number of data points for that analyte. A discussion summarizing the results of project completeness and any limitations on the use of the data will be described in the report.

The results will be presented in text of the Data Comparability Report. Data gaps will be evaluated if requested by USACE/EPA. The report will discuss the completeness of the planned and collected data and the affect on the data objective of evaluating the accuracy of the CPG's data.

Sensitivity – Data results will be compared to project action limits provided on Worksheet No.15. A discussion summarizing any conclusions about sensitivity of the analyses will be presented, and any limitations on the use of the data will be described in the report.

Reconciliation – The PQLGs presented in Worksheet No.12 will be examined to determine if the objectives were met. This examination will include a combined overall assessment of the results of each analysis pertinent to an objective. Each analysis will first be evaluated separately in terms of major impacts observed from data validation, data quality indicators and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be

determined. Based on the quality determined, the usability of the data for each analysis will be determined. Based on the combined usability of the data from all analyses for an objective, it will be determined if the PQLG was met and whether project goals were achieved. As part of the reconciliation of each objective, conclusions will be drawn and any limitations on the usability of any of the data will be described.

The following equations will be used:

1. To calculate split sample precision: $RPD = 100 * 2 |X1 - X2| / (X1 + X2)$

   where X1 and X2 are the reported concentrations for each duplicate or replicate

2. To calculate split data completeness:

% Completeness = V/n * 100 - where V= number of measurements judged valid; n = total number of measurements made and

% Completeness = C/x * 100 - where C= number of samples collected; x = total number of measurements planned

The investigation results will be presented in table and figures and in the text of the Data Comparability Report. Data gaps will be evaluated if requested by USACE/EPA. The report will discuss the completeness of the planned and collected data and the affect on the data objective of evaluating the accuracy of the CPG's data.

References:

CDM Federal Programs Corporation (CDM). 2010. *Final Quality Assurance Project Plan Physical Water Column Monitoring and Generic Information for Upcoming Tasks*. March 10.

CDM. 2010. *Final Quality Assurance Project Plan Addendum No. 5. Fish Tissue Analysis.* August 24.

Malcolm Pirnie and Battelle Duxbury Operations. 2009. *Oversight Quality Assurance Project Plan. Biological Sampling, Community Surveys, and Toxicity and Bioaccumulation Testing*. August.

Windward Environmental LLC (Windward). 2009. Lower Passaic River Restoration Project. *Quality Assurance Project Plan: Surface Sediment Chemical Analyses and Benthic Invertebrate Toxicity and Bioaccumulation Testing*. October 2.

Windward. 2011. Lower Passaic River Restoration Project. Lower Passaic River Study Area RI/FS. *Caged Bivalve Study Addendum to the Quality Assurance Project Plan. Surface Sediment Chemical Analyses and Benthic Invertebrate Toxicity and Bioaccumulation Testing*. Draft. February 8.

Appendix K

(Appendix K, the Laboratory Standard Operating Procedures

is included in the Oversight QAPP

Addendum No. 5, Fish Tissue Analysis.

Only the revised PAH-SIM SOP is included herein as Appendix L)

Appendix L

Columbia Analytical Services SOP

for Polycyclic Aromatic Hydrocarbons by

GC/MS Selective Ion Monitoring,

EPA Method 8270C SIM

STANDARD OPERATING PROCEDURE

POLYCYCLIC AROMATIC HYDROCARBONS BY GC/MS SELECTIVE ION MONITORING

EPA Method 8270C SIM

SOC-8270P
Revision 7
February 14, 2008

Approved By:

Supervisor		2/14/08 Date
QA Manager		2-14-08 Date
Laboratory Manager		2/14/08 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: CO Date: 4/14/09
Initials: CO Date: 4/5/10
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

POLYCYCLIC AROMATIC HYDROCARBONS BY GC/MS SELECTIVE ION MONITORING
Method 8270C SIM

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Semi-Volatile Organic Compounds in water, soil, and tissue matrices using EPA Method 8270C SIM. This procedure may also be applicable to various miscellaneous waste samples. Table 1 lists compounds that may be determined by this method and the standard method reporting limits (MRLs) and method detection limits (MDLs) in water, soil, and tissue. Table 1B lists additional low-level MRLs and MDLs in water, soil, and tissue. Alkylated PAHs listed in Table 1C may also be determined using this procedure. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The MDLs that have been achieved are listed in the tables however, the MDLs may change as MDL studies are repeated.

1.2. The procedure is intended for samples containing trace-level amounts of target compounds. Samples containing high concentrations of target analyte will not be analyzed undiluted. Extracts may be screened using GC/FID to estimate the hydrocarbon content and concentrations of individual polynuclear aromatic hydrocarbons (PAHs). Samples containing PAHs in excess of five times the high calibration standard will be diluted prior to analysis. All MRLs will be adjusted in accordance with this dilution. Therefore, samples containing high levels of PAHs will not be analyzed to achieve the optimum MRLs for the analysis.

1.3. This procedure can be used to quantitate most neutral organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone phase. This procedure is optimized for the analysis of polynuclear aromatic hydrocarbons.

1.4. Other compounds than those listed in Tables 1 and 1A may be analyzed. However, analytes not summarized in Table 1 have not been validated with a method detection limit study. Therefore, the lab will not use this procedure to analyze for non-routine analytes unless a similar analyte has been validated with a MDL study. As a general rule, the MRL for these compounds will equal the MRL of a similar compound in the routine analyte list. Results will not be reported below this estimated MRL.

2. METHOD SUMMARY

2.1. This method provides Gas Chromatography/Mass Spectrometry (GC/MS) conditions for the detection of Semi-volatile Organic Compounds. Prior to the use of this method, water

samples are extracted using either method 3520 (SOP EXT-3520) or 3535 (SOP EXT-3535) and soil/solid samples are extracted using 3541 (SOP EXT-3541).

2.2. All soil, tissue, and colored water extracts will be cleaned using method 3630 (silica gel cleanup, SOP SOC-3630) prior to analysis. In cases where project-specified analytes are not amenable to silica gel cleanup, gel permeation chromatography (SOP SOC-3640) will be used for cleanup prior to analysis.

2.3. An aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass selective detector in the selective ion mode. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing mass spectra of analytes with mass spectra of reference materials. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence The sequence begins with injection of Decafluorotriphenylphosphine (DFTPP) acquired in full scan mode followed by initial calibration standard(s) acquired in SIM mode. Once calibrated, a CCV is evaluated and extracts can be run. The sequence ends after 12 hours based on the DFTPP acquisition time.

3.2. **Laboratory Control Sample (LCS)** - In the LCS analysis, predetermined quantities of standard solutions of all analytes are added to a blank matrix prior to sample extraction and analysis. The purpose of the LCS is to monitor analytical control for the sample batch. Percent recoveries are calculated for each of the analytes.

3.3. **Matrix Spike/Duplicate Matrix Spike Analysis** - In the matrix spike analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, then spiked and analyzed. Percent recoveries are calculated for each of the controlled analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.4. **Standard Curve** - A standard curve is a plot of concentrations of a known analyte standard versus the instrument response to the analyte.

3.5. **Surrogate** - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in

environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and matrix spikes prior to extraction. Percent recoveries are calculated for each surrogate.

3.6. **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.7. **Continuing Calibration Verification Standard (CCV)** - A mid-level standard injected into the instrument at specified intervals and is used to verify the validity of the initial calibration.

3.8. **Second Source Verification Standard or Independent Verification Standard (SSV or ICV)** - A mid-level standard injected into the instrument after the calibration curve from a different source than the standards in the curve and is used to verify the validity of the initial calibration.

3.9. **Selective Ion Monitoring (SIM)** – Mass spectrometry technique where ions resulting from fragmentation are selectively monitored, therefore excluding other ions. The technique enhances sensitivity as compared to full scan analysis. Because the analysis results in significantly less mass spectral information, this gain in sensitivity is made at the expense of analyte selectivity. Therefore, the use of SIM results in significantly lower instrument detection limits, but increases the uncertainty associated with the analysis.

4. INTERFERENCES

4.1. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples. Corrective action should be taken to eliminate the interferences.

4.2. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of an instrument blank to check for carryover.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS

Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. This method uses Methylene Chloride, a known human carcinogen. Viton brand gloves should be used while rinsing, pouring or transferring the solvent

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Certified clean containers should be purchased for sample collection. Alternatively, containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Water and soil samples should be iced or refrigerated at 4 ± 2°C from time of collection until extraction. Tissue samples are stored frozen until extraction.

6.3. Water samples must be extracted within 7 days. Soil samples must be extracted within 14 days. Holding times for tissues are typically defined by project specifications, otherwise tissue samples may be held frozen up to one year before extraction. Extracts are stored at -10°C and must be analyzed within 40 days following extraction.

7. APPARATUS AND MATERIALS

7.1. Gas Chromatograph/Mass Spectrometer System

7.1.1. Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source. An injector capable of large volume injection must be attached to the GC system. Optic 2 and Optic 3 systems are recommended.

7.1.2. Column: 5% Dipenyl, 95% Dimethyl Polysiloxane - 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column or equivalent. Recommended: Restek XTI-5 with Integra-guard, catalog #12223-124.

7.1.3. Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode, and capable of operating in the SIM mode.

7.1.4. GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points for each compound of interest and achieves acceptable tuning performance criteria may be used.

7.1.5. Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits.

7.1.6. Instrumental systems are identified as follows:

Instrument I.D.	Analytical System	Routine Matrix
MS11	6890/5973	Water/Soil
MS14	6890/5973	Tissue
MS06	5890/5972	Water/Soil (overflow capacity)*
MS17	6890/5973	Soil/Tissue

* MDL studies are not maintained for overflow capacity instrumentation. Prior to any sample analyses on this instrument, MDL studies for each matrix and preparation method will be analyzed.

7.2. Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Solvents: Acetone, methylene chloride, methanol, and other appropriate solvents. Solvents must be of sufficient purity to permit usage without lessening the accuracy of the determination or introducing interferences.

8.2. Stock Standard Solutions

8.2.1. Commercially prepared stock standards are typically used when available at a concentration of 100 ug/ml or more. They must be A2LA or ISO9000 certified by the manufacturer. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions at a concentration of 1000 µg/ml by dissolving 0.0100 g of reference material in methylene chloride or other suitable solvent and diluting to volume in a 10mL volumetric flask. Larger volumes can be used at the convenience of the analyst.

When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8.2.2. Transfer stock standard solutions into amber Teflon-sealed crimp top autosampler vials. Store at -10°C and protect from light, or store as recommended by the manufacturer. Standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3. Unopened stock standards and neat materials have an expiration date equal to the manufacturer's recommendation. Neat material that does not have a manufacturer's recommended expiration date should be replaced after three years. Stock standard solutions received in sealed ampules with manufacture expiration dates in excess of 1 year have an expiration date of 1 year from the date of opening the sealed ampule.

8.2.4. For the alkylated PAH option, a PAH-ALKH SRM is used. This SRM is a petroleum crude oil which has been cleaned up using GPC and silica gel cleanup procedures. All of the homologs are present in the SRM.

8.3. Internal Standard Solutions - The internal standards are naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See Table 3 for corresponding compounds). The nominal concentration of the standard is 20 ng/µL. See Table 5 for standard preparation instructions. Each 1 ml of sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 0.2 ng/µL of each internal standard. Store at -10°C or less when not being used. When using premixed certified solutions, store according to the manufacturer's recommendations.

8.4. GC/MS Tuning Standard - A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP). The standard should also contain 50 ng/µL of pentachlorophenol and benzidine to verify injection port inertness and GC column performance. This injection is acquired in full scan mode and evaluated in accordance with the method specified criteria. Store at -10°C or less when not being used, or store according to the manufacturer's recommendations.

8.5. Calibration Standards

8.5.1. Prepare an intermediate surrogate standard by diluting 40uL of the 5000ppm stock to 2.0mL in DCM, resulting in 100ug/mL. An intermediate standard is prepared to combine the PAHs and surrogates into a standard that is used to prepare the calibration standards. See Table 5 for preparation instructions.

8.5.2. A minimum of six initial calibration standards should be prepared from stock solutions (note that a seven point calibration is recommended). One of the calibration standards should be at a concentration at or below the method reporting limit. The others should correspond to the range of concentrations found in

samples, but should not exceed the working range of the GC/MS system. Each 1 ml aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis.

8.5.3. All calibration standards should be stored at -10°C or less and should be freshly prepared from stocks every 365 days, or sooner if check standards indicate a problem.

8.5.4. The following calibration standards are recommended: 0.002 ng/µl, 0.004 ng/µl, 0.008 ng/µl, 0.02 ng/µl, 0.1 ng/µl, 0.2 ng/µl, 0.4 ng/µl, 1.0 ng/µl, 1.6 ng/µl and 2.0 ng/µl. See Table 5 for preparation instructions.

8.5.5. The independent calibration verification (ICV) standard is prepared at a nominal 0.4 ng/µL concentration from stock solutions (see Table 5). The ICV is prepared at the time of initial calibration and can be stored at 4°C ± 2°C.

8.5.6. The daily calibration standard (CCV) is prepared at a nominal 0.4 ng/µL concentration from stock solutions (see Table 5). The CCV is prepared weekly and can be stored at 4°C ± 2°C.

8.6. QC Standards

8.6.1. Surrogates: Prepare a working spiking solution in methanol containing Fluorene-d10, Fluoranthene-d10, and Terphenyl-d14 at 100 ng/µL. This solution may be combined with the surrogate solution used to monitor analyses for 8270 full list. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used.

8.6.2. Matrix Spike Standards: Prepare a working spiking solution in methanol containing all analytes of interest ("full list spike"). All analytes are prepared at 25 ng/µl. Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.4. Mass Spectrometer

9.4.1. For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

9.4.2. MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

9.4.3. Tune the MS as needed to result in consistent and acceptable performance while meeting the required ion abundance criteria.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8270C, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water, soil, tissue and waste samples are prepared using the appropriate extraction and cleanup methods (refer to SOPs) and may be screened by GC/FID (see SOP SOC-SCR).

11.1.2. An appropriate cleanup procedure may be performed depending on the sample matrix and target analytes. Perform cleanups on all soil, tissue, and colored water extracts using method 3630 (silica gel cleanup, SOP SOC-3630) prior to analysis. In cases where project-specified analytes are not amenable to silica gel cleanup, perform method 3640 GPC cleanup (SOP SOC-3640) prior to analysis.

11.1.3. Following sample preparation, sample extracts are then transferred to the extract cold storage unit. Extracts must be analyzed within 40 days of extraction.

11.2. The recommended GC/MS operating conditions:

Ion dwell time:	10 – 50 msec per ion
Initial temperature:	40°C, hold for 1 minutes
Temperature program:	40-140°C at 30°C/min hold for 0 minutes
Temperature program:	140-270°C at 10°C/min hold for 4 minutes
Final temperature:	270-320°C at 20°/min, hold for 1.10 minutes
Injector temperature:	320°C
Detector interface temp:	300°C
Injector:	splitless, electronic pressure control with pulse
Sample volume:	5.0 µL
Carrier gas:	helium at 1.2ml/min (constant flow)

11.3. Selected Ion Acquisition

11.3.1. Determine the ions to be monitored for the compounds of interest. Refer to Table 2 for characteristic ions. At a minimum, 2 ions should be monitored for each compound, and 3 monitored for compounds with more complex fragmentation patterns. Set the SIM windows in order to monitor the correct ions at the correct time, based on chromatographic elution of the compounds. This can be setup by analyzing a standard using a full scan analysis and using the GC conditions of the SIM analysis. This analysis will give retention time and spectral information for determining the location of start times for the SIM groups or windows. This is often referred to as a "locator" analysis.

11.3.2. Select the dwell times to be used for each group of ions to be monitored. Dwell times should be selected in order to provide a sufficient number of measurements

across the chromatographic peak to accurately define the peak shape. Too few measurements across the peak will result in poor definition of the peak and subsequently result in poor accuracy and precision of results. Too many measurements across the peak may result in inconsistent detector behavior over the calibration range. Significant differences in dwell times may also affect sensitivity. Typical dwell times are listed in section 11.2.

11.4. Initial Calibration

Refer to the SOP for *Calibration of Instruments for Organics Chromatographic Analysis* (SOC-CAL) for general calibration procedures. The calibration procedure and options chosen must follow SOP SOC-CAL and this SOP. In general, the calibration procedure is as follows:

11.4.1. Prior to calibration, analyze the GC/MS tuning standard using instrument conditions used for calibration. Obtain the spectrum for evaluation using one of the following options:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.
- Use one scan at the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.
- Use one scan either directly preceding or following the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.
- Use the average across the entire peak up to a total of 5 scans. Peak integration must be consistent with standard operating procedure. If the peak is wider than 5 scans, the tune will consist of the peak apex scan and the two scans immediately preceding and following the apex. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not

subtract part of the tune peak or part of any other peak eluting close to the tune peak.

11.4.2. Evaluate the spectrum obtained for DFTPP against the tuning criteria in Table 4 or 4A (dependent upon instrumentation). The GC/MS must meet the DFTPP ion abundance criteria prior to further analyses. Pentachlorophenol must be present at the normal response, with no visible peak tailing, as demonstrated by the peak tailing factors.

The acceptance criteria for the peak tailing factor for pentachlorophenol is < 5.0. If excessive tailing or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to remove the first 15-30 cm of the GC column. If hardware tuning criteria cannot be met, the source may need cleaning, filaments replaced or other maintenance.

11.4.3. The internal standards should permit most of the components of interest in the chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Refer to Table 3 for internal standards and corresponding analytes assigned for quantitation. Use the base peak ion from the specific internal standard as the primary ion for quantitation (See Table 2). If interferences are noted, use the next most intense ion as the quantitaion ion (i.e. for acenaphthene-d_{10}, use 162 m/z for quantitation).

11.4.4. Analyze 5.0 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 2). Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for compound being measured.
A_{is} = Area of the characteristic ion for specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

11.4.5. A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. The minimum acceptable average RF for these compounds is 0.10. If they are not acceptable, perform GC maintenance (see section 9).

11.4.6. The percent relative standard deviation (%RSD) should be less than 15% for each compound. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RSD = relative standard deviation.
RF = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - \overline{RF})^2}{N - 1}}$$

where:

RF_i = RF for each of the 5 calibration levels
N = Number of RF values (i.e., 5)

11.4.7. Linearity - If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.4.8. In those instances where the %RSD for one or more analytes exceeds 15%, the initial calibration may still be acceptable if the following conditions are met:

11.4.8.1. The mean of the RSD values for all analytes in the calibration is $\leq$ 15% and the %RSD does not exceed 30% for any compound.

11.4.8.2. The mean RSD criteria applies to all target analytes in the calibration standards, regardless of whether or not they are of interest for a specific project.

11.4.8.3. The data user must be supplied with a list of compounds which exceed 15% RSD and the result of the mean RSD calculation. For tier III and higher deliverables, an initial calibration summary may be used.

11.4.9. If all of the conditions in Section 11.4.8 are met, then the average response factor may be used to determine sample concentrations as described in Section 11.4.4.

11.4.10. When analysis for alkylated PAHs is to be performed, follow the calibration procedure given in Appendix A.

11.4.11. If the RSD of any target analyte is greater than 15%, refer to SOP SOC-CAL, section 11.2.3 and sections 7.5.2 and 7.5.3 in Method 8000B for additional calibration options. One of the options must be applied to initial calibration in this situation, or a new initial calibration must be performed.

11.4.12. Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. For each compound of interest, the calculated value must be ± 20% of the true value for the initial calibration to be valid.

11.5. Continuing Calibration

11.5.1. Following an acceptable tune, a calibration standard, or standards, at mid-concentration containing all PAH analytes, and all required surrogates, must be analyzed every 12 hours during analysis.

11.5.2. For each daily calibration, a system performance check must be made. For each compound in the daily calibration standard, a minimum response factor of 0.10 must be obtained. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

11.5.3. If the percent drift for each compound of interest is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% drift) for any one compound, corrective action must be taken. Problems similar to those listed in 11.5.2 could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new initial calibration must be generated. This criterion must be met before sample analysis begins.

Calculate the percent drift using:

$$\%\,Drift = \frac{C_I - C_c}{C_I} \times 100$$

where:

C_I = Compound standard concentration.
C_c = Measured concentration using selected quantitation method.

11.5.4. When analysis for alkylated PAHs is to be performed, follow the calibration procedure given in Appendix A.

11.5.5. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as required. If the EICP area for any of the internal standards changes by a factor of two (50% to 200%) from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as appropriate.

When corrective action is taken, reanalysis of samples analyzed while the system was malfunctioning is required. Update the reference spectra and retention times in the quantitation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quantitation of subsequent analyses.

11.5.6. A blank (method blank, GPC blank, or solvent blank) should be analyzed after the CCV to prove the system is free of contaminants. If contaminants are found in a method blank or GPC blank, then a solvent blank should be ran to help isolate the source of contamination.

11.6. GC/MS Analysis

11.6.1. Evaluate FID screen and make proper dilution (See SOP SOC-SCR).

11.6.2. Spike the 1 ml extract obtained from sample preparation with 10 µL of the internal standard solution just prior to analysis. Use the same operating conditions as were used for initial calibration.

11.6.3. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 0.2ng/µL of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

11.6.4. Store the extracts at -10°C or less, protected from light in vials equipped with unpierced Teflon lined septa. Archive extract in freezer for 3 months, or longer if required by client, after analysis in the instrument/date specific storage boxes.

12. QA/QC REQUIREMENTS

12.1. In addition to instrument criteria for calibration, the ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be validated before analysis of samples begin, or whenever significant changes to the procedures have been made. To do this, four deionized water samples are spiked with each target analyte, extracted, and analyzed. Refer to Method 8270C Section 8.3 for this requirement and acceptance criteria.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze.

12.2.2. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP *for The Determination of Method Detection Limits and Limits of Detection (ADM-MDL).*

12.2.3. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the *SOP for Sample Batches*. In general, these include:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants.

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Attachment A. If the lab control sample (LCS) fails acceptance limits for any of the compounds, the analyst must evaluate the system and calibration. Corrective action must be taken.

12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = % recovery of the MS
R2 = % recovery of the DMS

The acceptance limits for the MS/DMS are given in Attachment A. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken.

12.3.4. The acceptance limits for the surrogates are given in Attachment A. If any surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be taken. The sample should be re-analyzed if instrument factors (calibration, injection port) are suspected. If not, re-extraction and re-analysis is required, except in cases of high recovery and no positive hits in the sample for the analyte class represented by the particular surrogate.

12.3.5. The acceptance criteria listed in Attachment A are current criteria, but are subject to change as control limits are updated.

12.3.6. Additional QA/QC measures include control charting of QC sample results.

12.3.7. Corrective action – When a data quality objective is not met, the initial corrective action will include a review of the raw data for potential calculation and/or integration errors. If this review does not correct the problem, the following corrective actions will be performed.

12.3.7.1. Method Blank – No target analyte should be detected in a method blank at or above the method reporting limit (1/2 the MRL for DoD projects). If target analytes are detected in the method blank, the sample data must be reviewed for possible laboratory contribution. Detections of target analytes greater than the MRL require a Nonconformity and Corrective Action Report (NCAR). A decision to reextract the associated samples will depend on the level of the contamination, data quality implications, and the intended use of the data. At a minimum, all positive detections in the associated samples that are not more than 20X the concentration in the blank will be qualified with a "B". Also, as part of the corrective action, the problem will be discussed with the appropriate sample prep personnel in an effort to identify the contamination source.

12.3.7.2. Laboratory Control Sample – The analysis should include a full list LCS spike. All target analytes will be evaluated. The following cases require corrective action:

- If any analytes do not meet acceptance criteria, the analytical batch should be considered out of control for that analyte. Corrective action may include reinjection to verify the result. If the result is confirmed, a NCAR will be filed and the problem investigated to determine that cause. A decision to reextract the associated samples will depend on the data quality implications and the intended use of the data, and should involve the Project Chemist and client. If reextraction is not feasible, all reported results for that analyte will be qualified and the implications will be discussed in the case narrative.
- In cases where a result is outside the upper control criterion, corrective action is only required if that analyte was also detected in field samples. The associated samples with positive results should be reextracted. In cases where a result is outside the lower control criterion, the associated samples should be reextracted. If reextraction is not feasible, all reported results for that analyte will be qualified and the data quality implications will be discussed in the case narrative. Hoever, investigation into the cause of the failure should still be performed.

12.3.7.3. Matrix Spike and Duplicate Matrix Spike Samples – If a recovery is outside of control criteria, review the consistency between the two analyses. If the result is supported between the two analyses, the outlier can be attributed to matrix inteference. Do not reanalyze the extract. If the results do not support each other, reanalyze the extracts to verify the results. If the results confirm, review the LCS recovery and take corrective action accordingly. If the LCS recovery is acceptable, flag the matrix spike data and discuss potential data quality implications in the case narrative.

12.3.7.4. Relative Percent Difference – For MS/DMS or LCS/DLCS, no corrective action is required based on RPD data alone. However, the data should be reviewed for information that will help determine if the RPD problem is the result of a sample specific issue (e.g., the DMS was concentrated to dryness), or if the problem is representative of the entire analytical batch. When the problem is apparently universal to the batch, a NCAR will be filed and the batch will be reextracted. If results of the reextraction confirm the original analyses of the field samples, the original data is reported and the RPD problems are discussed in the case narrative. If results of the reextraction confirm a problem in the original data, only the reextracted data is reported.

12.3.7.5. Surrogates – Corrective action includes reinjection to verify the result. If the result is confirmed, a NCAR will be filed and the sample will be reextracted. If the reextraction confirms the original results are biased due to matrix interferences, report the original data. If reextraction is not feasible, the surrogate will be qualified and the data quality implications will be discussed in the case narrative.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the sample analysis. The characteristic ions from the reference mass spectrum are defined to be the ions monitored in the SIM mode and typically are the two or three ions of greatest relative intensity. Compounds are identified as present when the criteria below are met.

13.1.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.1.2. The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

13.1.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

13.1.4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the 2 peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.1.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When the gas chromatographic peaks appear to represent more than one component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification. When analytes coelute, the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

13.1.6. Evaluate internal standard areas in each sample. If the area in the sample is less than 50% or greater than 200% the area of in the CCV, corrective action must be taken. Depending on the analysis, this corrective action may include reinjection or dilution of the extract followed by reinjection.

13.2. Tentatively identified compounds (TICs) cannot be reported using this method.

13.3. Quantitation and Calculations

13.3.1. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions. When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: C_{ex} = the concentration in the sample extract (ppm);
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppm, of internal standard added
$\overline{RF_x}$ = the average response from the initial calibration.

13.3.2. The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples: $Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$

Nonaqueous Samples: $Concentration\ (ug/Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$

Where
Cex = Concentration in extract in ng/mL
Vf = Final volume of extract in mL
D = Dilution factor
Vs = Volume of sample extracted, liters
W = Weight of sample extracted in grams.

13.4. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details.

13.5. Reporting

Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument, date, and client-specified report requirements (when specified). This compilation is then transferred to a file which the Stealth reporting system uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

14. CONTINGENCIES FOR HANDLING OUT-OF- CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the *SOP for Corrective Action* (ADM-NCAR) for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the *SOP for Report Generation* (ADM-RG) or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for *the Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses Methylene Chloride and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

18. TRAINING OUTLINE

18.1. The following items provide guidelines for training analysts.

18.1.1. Review applicable literature (method references, etc.) and this SOP. Review the MSDS for all chemicals used in the analysis.

18.1.2. Observe the procedure as performed by an experienced analyst at least three times.

18.1.3. Assist in the procedure under the guidance of an experienced analyst for at least one month, preferably three months. During this training period, the analyst is expected to progress from a role of assisting to a role of performing the procedure with minimal oversight.

18.2. Following this training period, the analyst is expected to complete an Initial Precision and Recovery (IPR) study as described in Section 12. Documentation of the IPR study should be forwarded to the analyst's training file.

19. REFERENCES

19.1. *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry*, Method 8270C, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update III, December 1996.

19.2. Exxon Valdez Spill Assessment Procedure EV89-2, Revision 2.0, June 1989.

19.3. Standard Methods Manual for Environmental Sampling and Analysis in San Francisco Bay, US Army Corps of Engineers, November 1992 Draft, Volume 2 of 3.

19.4. CAS SOPS

19.4.1. Continuous Liquid-Liquid Extraction, EXT-3520

19.4.2. Solid Phase Extraction, EXT-3535

19.4.3. Automated Soxhlet Extraction, EXT-3541

19.4.4. Silica Gel Cleanup, SOC-3630

19.4.5. Gel Permeation Chromatography, SOC-3640

TABLE 1

Target Analytes, Method Reporting Limits, and Method Detection Limits (Standard Level)

	WATER µg/L (ppb)		SOIL µg/Kg Dry Weight (ppb)		Tissue µg/Kg Wet Weight (ppb)	
Analytes	**MRL**	**MDL**	**MRL**	**MDL**	**MRL**	**MDL**
Naphthalene	0.02	0.0030	5	0.37	5	0.40
2-Methylnaphthalene	0.02	0.0023	5	0.39	5	0.44
1-Methylnaphthalene	0.02	0.0035	5	0.31	5	0.30
Biphenyl	0.02	0.0024	5	0.40	5	0.17
2,6-Dimethylnaphthalene	0.02	0.0022	5	0.36	5	0.41
Acenaphthylene	0.02	0.0034	5	0.24	5	0.069
Acenaphthene	0.02	0.0044	5	0.23	5	0.11
Dibenzofuran	0.02	0.0046	5	0.59	-	-
2,3,5-Trimethylnaphthalene	0.02	0.0050	5	0.21	5	0.24
Fluorene	0.02	0.0038	5	0.50	5	0.15
Phenanthrene	0.02	0.0050	5	0.75	5	0.36
Anthracene	0.02	0.0036	5	0.47	5	0.065
1-Methylphenanthrene	0.02	0.0041	5	0.28	5	0.082
Fluoranthene	0.02	0.0044	5	0.61	5	0.090
Pyrene	0.02	0.0035	5	0.37	5	0.098
Benz(a)anthracene	0.02	0.0026	5	0.48	5	0.066
Chrysene	0.02	0.0034	5	0.25	5	0.076
Benzo(b)fluoranthene	0.02	0.0023	5	0.25	5	0.070
Benzo(k)fluoranthene	0.02	0.0025	5	0.15	5	0.056
Benzo(e)pyrene	0.02	0.0040	5	0.18	5	0.062
Benzo(a)pyrene	0.02	0.0043	5	0.14	5	0.081
Perylene	0.02	0.0050	5	0.32	-	-
Indeno(1,2,3-cd)pyrene	0.02	0.0026	5	0.16	5	0.064
Dibenz(a,h)anthracene	0.02	0.0025	5	0.28	5	0.059
Benzo(g,h,i)perylene	0.02	0.0029	5	0.64	5	0.073

TABLE 1B

Target Analytes, Method Reporting Limits, and Method Detection Limits (Low Level)

	WATER ng/L (ppt)		SOIL µg/Kg Dry Weight (ppb)		Tissue µg/Kg Wet Weight (ppb)	
Analytes	**MRL**	**MDL**	**MRL**	**MDL**	**MRL**	**MDL**
Naphthalene	3.4	0.76	1.0	0.16	1	0.40
2-Methylnaphthalene	3.4	0.65	1.0	0.11	1	0.44
1-Methylnaphthalene	3.4	0.45	-	-	0.5	0.30
Biphenyl	3.4	0.14	-	-	0.5	0.17
2,6-Dimethylnaphthalene	3.4	0.11	-	-	0.5	0.41
Acenaphthylene	3.4	0.24	0.5	0.073	0.5	0.069
Acenaphthene	3.4	0.27	0.5	0.093	0.5	0.11
Dibenzofuran	3.4	0.67	0.5	0.12	0.5	-
2,3,5-Trimethylnaphthalene	3.4	0.18	-	-	0.5	0.24
Fluorene	3.4	0.21	0.5	0.086	0.5	0.15
Phenanthrene	3.4	1.8	0.5	0.12	0.5	0.36
Anthracene	3.4	0.20	0.5	0.089	0.5	0.065
1-Methylphenanthrene	3.4	0.13	-	-	0.5	0.082
Fluoranthene	3.4	0.24	0.5	0.15	0.5	0.090
Pyrene	3.4	0.27	0.5	0.17	0.5	0.098
Benz(a)anthracene	3.4	0.25	0.5	0.21	0.5	0.066
Chrysene	3.4	0.29	0.5	0.013	0.5	0.076
Benzo(b)fluoranthene	3.4	0.24	0.5	0.11	0.5	0.070
Benzo(k)fluoranthene	3.4	0.19	0.5	0.094	0.5	0.056
Benzo(e)pyrene	3.4	0.19	-	-	0.5	0.062
Benzo(a)pyrene	3.4	0.27	0.5	0.083	0.5	0.081
Perylene	-	-	-	-	0.5	0.064
Indeno(1,2,3-cd)pyrene	3.4	0.28	0.5	0.083	0.5	0.064
Dibenz(a,h)anthracene	3.4	0.25	0.5	0.090	0.5	0.059
Benzo(g,h,i)perylene	3.4	0.15	0.5	0.065	0.5	0.073

TABLE 1C

Additional Analytes
Target Analytes and Method Reporting Limits

	WATER µg/L (ppb)	SOIL µg/Kg Dry Weight (ppb)	Tissue µg/Kg Wet Weight (ppb)
Analytes	**MRL**	**MRL**	**MRL**
C2-Naphthalenes	0.02	5	5
C3-Naphthalenes	0.02	5	5
C4-Naphthalenes	0.02	5	5
C1-Fluorenes	0.02	5	5
C2-Fluorenes	0.02	5	5
C3-Fluorenes	0.02	5	5
C1-Dibenzothiophenes	0.02	5	5
C2-Dibenzothiophenes	0.02	5	5
C3-Dibenzothiophenes	0.02	5	5
C1-Phenanthrenes/Anthracenes	0.02	5	5
C2-Phenanthrenes/Anthracenes	0.02	5	5
C3-Phenanthrenes/Anthracenes	0.02	5	5
C4-Phenanthrenes/Anthracenes	0.02	5	5
C1-Fluoranthenes/Pyrenes	0.02	5	5
C2-Fluoranthenes/Pyrenes	0.02	5	5
C3-Fluoranthenes/Pyrenes	0.02	5	5
C1-Chrysenes	0.02	5	5
C2-Chrysenes	0.02	5	5
C3-Chrysenes	0.02	5	5
C4-Chrysenes	0.02	5	5

TABLE 2
TARGET COMPOUNDS AND CORRESPONDING PRIMARY AND SECONDARY IONS

Compound	Approximate Retention Time (min)	Primary Ion	Secondary Ion
Naphthalene-d 8 (I.S.)	5.86	136	68
Naphthalene	5.87	128	127
2-Methylnaphthalene	6.62	141	142
1-Methyl naphthalene	6.74	141	142
Biphenyl	7.18	154	153
2,6-Dimethylnaphthalene	7.37	156	155
Acenaphthlene-d 10 (I.S.)	7.95	164	162
Acenaphthylene	7.75	152	153
Acenaphthlene	8.00	154	153
Dibenzofuran	8.22	168	139
2,3,5-Trimethylnaphthalene	8.53	170	155
Fluorene	8.70	166	165
Phenanthrene-d 10 (I.S.)	10.10	188	94
Dibenzothiophene	9.95	184	152
Phenanthrene	10.13	178	179
Anthracene	10.20	178	176
1-Methylphenanthrene	11.10	192	150
Fluoranthene	11.99	202	203
Chrysene-d 12 (I.S.)	14.51	240	236
Pyrene	12.35	202	203
Benz (a) anthracene	14.48	228	226
Chrysene	14.57	228	226
Perylene-d 12 (I.S.)	18.48	264	260
Benzo(b)fluoranthene	17.50	252	126
Benzo(k)fluoranthene	17.56	252	126
Benzo(e)pyrene	18.22	252	126
Benzo(a)pyrene	18.33	252	126
Perylene	18.52	252	126
Indeno (1,2,3-cd) pyrene	20.42	276	277
Dibenz (a, h) anthracene	20.47	278	276
Benzo(g,h,i)perylene	20.86	276	277
Fluorene-d10 (surr.)	8.67	176	175
Fluoranthene-d10 (surr.)	11.97	212	213
Terphenyl-d 14 (surr.)	12.63	244	122

TABLE 3

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

Naphthalene-d8	**Acenaphthene-d10**	**Phenanthrene-d10**
Naphthalene	Acenaphthene	Dibenzothiophene
2-Methylnaphthalene	Acenaphthylene	Phenanthrene
1-Methylnaphthalene	Dibenzofuran	Anthracene
Biphenyl	2,3,5-Trimethylnaphthalene	1-Methylphenanthrene
2,6-Dimethylnaphthalene	Fluorene	Fluoranthene
	Fluorene-d_{10} (surr.)	Fluoranthene-d_{10} (surr.)

Chrysene-d12	**Perylene-d12**
Pyrene	Benzo(b)fluoranthene
Benzo(a) anthracene	Benzo(k)fluoranthene
Chrysene	Benzo(e)pyrene
Terphenyl-d_{14} (surr.)	Benzo(a)pyrene
	Perylene
	Indeno (1,2,3-ccd)pyrene
	Dibenz(a,h)anthracene
	Benzo(g,h,i)perylene

TABLE 4
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA
for **5973** GC/MS systems

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	0-2% of mass 69
70	0-2% of mass 69
127	10-80% of 198
197	0-2% of 198
198	30-100% of 442 (alternate base)
199	5-9% of 198
275	10-60% of 198
365	1-50% of 442
441	0.01-100% of 443
442	30-100% of 198 (alternate base)
443	15-24% of 442

TABLE 4A
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA
for **5972** GC/MS systems

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	0-2% of mass 69
69	Present
70	0-2% of mass 69
127	25-75% of 198
197	0-1% of 198
198	100% Relative abundance, Base Peak
199	5-9% of 198
275	>0.75% of 198
365	1-50% of 442
441	0.01-99.99% of 443
442	40-110% of 198
443	15-24% of 442

TABLE 5

STANDARD PREPARATION

Internal Standard Working Standard

Parent Solution	Initial Concentration	Aliquot	Final Volume	Final Concentration	Solvent
AccuStandard Z-014J	4000 ug/mL	50 uL	10 mL	20 ug/mL	DCM

Initial Calibration Intermediate Standard

Parent Solution	Initial Concentration	Aliquot	Final Volume	Final Concentration	Solvent
Absolute PAH Mix	100 ug/mL	80 uL	2 mL	4 ug/mL	DCM
PAH Surr. Intermediate	100 ug/mL	80 uL	↓	4 ug/mL	↓

Initial Calibration Standards

Initial Cal. Intermediate Std. 4ug/mL	Internal Std Working Std 20ug/mL	Final Volume	Solvent	Final Concentration PAH	Final Concentration Int Std
5 uL	100 uL	10 mL	DCM	0.002 ug/mL	0.2 ug/mL
5 uL	50 uL	5 mL		0.004 ug/mL	
4 uL	20 uL	2 mL		0.008 ug/mL	
5 uL	10 uL	1 mL		0.02 ug/mL	
25 uL	↓	↓		0.1 ug/mL	
50 uL				0.2 ug/mL	
100 uL				0.4 ug/mL	
250 uL				1.0 ug/mL	
400 uL				1.6 ug/mL	
500uL			↓	2.0 ug/mL	↓

Independent Calibration Verification (ICV) Standard

Parent Solution	Initial Concentration	Aliquot	Final Volume	Final Concentration	Solvent
Cerilliant PAH Mix CSQ-4049	100 ug/mL	20 uL	5 mL	0.4 ug/mL	DCM
PAH Surr. Intermediate	100 ug/mL	20 uL	↓	0.4 ug/mL	↓
Int. Std Working Std	20 ug/mL	50 uL		0.2 ug/mL	

APPENDIX A

Procedure for quantifying alkylated homologs of polynuclear aromatic hydrocarbons

1. Analyze a PAH ICAL using the A_PAHK method. These acquisition parameters will acquire data for the parent PAHs as well as the homologs.

Process the data and update the calibration table. Go into EDIT COMPOUNDS which is under the INITCAL menu. Enter the peak areas from the parent compounds into the corresponding levels in the associated homologs calibration table as listed below.

PARENT	HOMOLOG
Naphthalene	C2-Naphthalenes
	C3-Naphthalenes
	C4-Naphthalenes
Fluorene	C1-Fluorenes
	C2-Fluorenes
	C3-Fluorenes
Dibenzothiophene	C1-Dibenzothiophenes
	C2-Dibenzothiophenes
	C3-Dibenzothiophenes
Phenanthrene	C1-Phenanthrenes/Anthracenes
	C2-Phenanthrenes/Anthracenes
	C3-Phenanthrenes/Anthracenes
	C4-Phenanthrenes/Anthracenes
Pyrene	C1-Fluoranthenes/Pyrenes
	C2-Fluoranthenes/Pyrenes
	C3-Fluoranthenes/Pyrenes
Chrysene	C1-Chrysenes
	C2-Chrysenes
	C3-Chrysenes
	C4-Chrysenes

Use the same Curve Fit for the Homolog as is used for the Parent compound. Use the average RF if the %RSD is less than 20%. Use a quadratic curve if the %RSD exceeds 20%.

2. Analyze the PAH-ALKH SRM. This SRM is a petroleum crude oil which has been cleaned up using GPC and silica gel cleanup procedures. All of the homologs are present in the SRM.

3. Process the SRM data against the ICAL. Review the SRM data in QEDIT and manually integrate the homologs using the example in the SOP book as a guide. Print a graphics report for each homolog.

4. Update the calibration table using the PAH CCV. Enter the average retention time for each of the homologs into the processing method.

5. Analyze the sample extracts and process the data against the ICAL. Using the SRM data as a guide, manually integrate the homologs. Print graphics reports for each sample hit.

Attachment A
QC Acceptance Criteria

SEMIVOLATILE ORGANICS ANALYSES							
Method	Prep Method	Matrix	Analyte		LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8270-SIM	3541	Soil	1-Methylnaphthalene		42-112	37-102	40
8270-SIM	3541	Soil	1-Methylphenanthrene		61-114	54-112	40
8270-SIM	3541	Soil	2,3,5-Trimethylnaphthalene		53-118	49-105	40
8270-SIM	3541	Soil	2,6-Dimethylnaphthalene		35-121	39-106	40
8270-SIM	3541	Soil	2-Methylnaphthalene		41-113	21-120	40
8270-SIM	3541	Soil	Acenaphthene		47-113	25-123	40
8270-SIM	3541	Soil	Acenaphthylene		46-115	33-115	40
8270-SIM	3541	Soil	Anthracene		53-116	23-134	40
8270-SIM	3541	Soil	Benz(a)anthracene		58-111	18-140	40
8270-SIM	3541	Soil	Benzo(a)pyrene		57-119	11-146	40
8270-SIM	3541	Soil	Benzo(b)fluoranthene		53-125	15-144	40
8270-SIM	3541	Soil	Benzo(e)pyrene		56-118	60-102	40
8270-SIM	3541	Soil	Benzo(g,h,i)perylene		43-122	13-135	40
8270-SIM	3541	Soil	Benzo(k)fluoranthene		54-123	21-131	40
8270-SIM	3541	Soil	Biphenyl		34-121	20-126	40
8270-SIM	3541	Soil	Carbazole		10-120	35-164	40
8270-SIM	3541	Soil	Chrysene		53-122	14-147	40
8270-SIM	3541	Soil	Dibenz(a,h)anthracene		37-126	14-133	40
8270-SIM	3541	Soil	Dibenzofuran		44-116	26-119	40
8270-SIM	3541	Soil	Dibenzothiophene		26-110	10-113	40
8270-SIM	3541	Soil	Fluoranthene		54-120	12-150	40
8270-SIM	3541	Soil	Fluorene		49-115	15-138	40
8270-SIM	3541	Soil	Indeno(1,2,3-cd)pyrene		43-119	11-132	40
8270-SIM	3541	Soil	Naphthalene		47-103	24-111	40
8270-SIM	3541	Soil	Perylene		58-119	18-123	40
8270-SIM	3541	Soil	Phenanthrene		52-111	15-138	40
8270-SIM	3541	Soil	Pyrene		53-120	12-152	40
8270-SIM	3541	Soil	Fluoranthene-d10 (Surr.)	10-141	NA	NA	NA
8270-SIM	3541	Soil	Fluorene-d10 (Surr.)	10-126	NA	NA	NA
8270-SIM	3541	Soil	Terphenyl-d14 (Surr.)	25-139	NA	NA	NA
8270-SIM	3510C/20C/35	Water	1-Methylnaphthalene		45-120	47-106	30
8270-SIM	3510C/20C/35	Water	1-Methylphenanthrene		55-146	35-154	30
8270-SIM	3510C/20C/35	Water	2,3,5-Trimethylnaphthalene		48-133	31-142	30
8270-SIM	3510C/20C/35	Water	2,6-Dimethylnaphthalene		34-124	15-149	30
8270-SIM	3510C/20C/35	Water	2-Methylnaphthalene		42-117	33-122	30
8270-SIM	3510C/20C/35	Water	Acenaphthene		56-119	46-126	30
8270-SIM	3510C/20C/35	Water	Acenaphthylene		55-123	41-136	30
8270-SIM	3510C/20C/35	Water	Anthracene		47-122	42-131	30
8270-SIM	3510C/20C/35	Water	Benz(a)anthracene		60-124	39-136	30
8270-SIM	3510C/20C/35	Water	Benzo(a)pyrene		42-136	24-145	30
8270-SIM	3510C/20C/35	Water	Benzo(b)fluoranthene		61-135	26-149	30
8270-SIM	3510C/20C/35	Water	Benzo(e)pyrene		58-133	30-144	30
8270-SIM	3510C/20C/35	Water	Benzo(g,h,i)perylene		47-132	18-140	30
8270-SIM	3510C/20C/35	Water	Benzo(k)fluoranthene		59-132	25-149	30
8270-SIM	3510C/20C/35	Water	Biphenyl		39-128	10-157	30
8270-SIM	3510C/20C/35	Water	Carbazole		27-177	42-147	30
8270-SIM	3510C/20C/35	Water	Chrysene		63-128	42-136	30
8270-SIM	3510C/20C/35	Water	Dibenz(a,h)anthracene		41-137	14-146	30
8270-SIM	3510C/20C/35	Water	Dibenzofuran		51-122	36-134	30
8270-SIM	3510C/20C/35	Water	Dibenzothiophene		50-139	70-130	30
8270-SIM	3510C/20C/35	Water	Fluoranthene		63-132	50-137	30
8270-SIM	3510C/20C/35	Water	Fluorene		58-124	48-130	30
8270-SIM	3510C/20C/35	Water	Indeno(1,2,3-cd)pyrene		44-131	16-143	30

SEMIVOLATILE ORGANICS ANALYSES						
Method	Prep Method	Matrix	Analyte	LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8270-SIM	3510C/20C/35	Water	Naphthalene	49-113	40-124	30
8270-SIM	3510C/20C/35	Water	Perylene	28-148	14-136	30
8270-SIM	3510C/20C/35	Water	Phenanthrene	60-123	55-127	30
8270-SIM	3510C/20C/35	Water	Pyrene	60-131	48-136	30
8270-SIM	3510C/20C/35	Water	1,4-Dioxane	57-111	54-106	30
8270-SIM	3510C/20C/35	Water	1,4-Dioxane-d8 (Surr.) 47-91	NA	NA	NA
8270-SIM	3510C/20C/35	Water	Fluoranthene-d10 (Surr.) 49-131	NA	NA	NA
8270-SIM	3510C/20C/35	Water	Fluorene-d10 (Surr.) 49-123	NA	NA	NA
8270-SIM	3510C/20C/35	Water	Terphenyl-d14 (Surr.) 37-140	NA	NA	NA
8270-SIM	3541	Tissue	1-Methylnaphthalene	47-116	36-110	40
8270-SIM	3541	Tissue	1-Methylphenanthrene	52-122	36-115	40
8270-SIM	3541	Tissue	2,3,5-Trimethylnaphthalene	50-116	58-125	40
8270-SIM	3541	Tissue	2,6-Dimethylnaphthalene	48-116	29-133	40
8270-SIM	3541	Tissue	2-Methylnaphthalene	43-116	37-117	40
8270-SIM	3541	Tissue	Acenaphthene	44-117	49-114	40
8270-SIM	3541	Tissue	Acenaphthylene	43-122	41-127	40
8270-SIM	3541	Tissue	Anthracene	46-124	48-123	40
8270-SIM	3541	Tissue	Benz(a)anthracene	45-135	46-129	40
8270-SIM	3541	Tissue	Benzo(a)pyrene	44-143	48-127	40
8270-SIM	3541	Tissue	Benzo(b)fluoranthene	49-138	44-125	40
8270-SIM	3541	Tissue	Benzo(e)pyrene	52-134	64-121	40
8270-SIM	3541	Tissue	Benzo(g,h,i)perylene	45-132	44-124	40
8270-SIM	3541	Tissue	Benzo(k)fluoranthene	49-141	47-127	40
8270-SIM	3541	Tissue	Biphenyl	48-115	54-124	40
8270-SIM	3541	Tissue	Carbazole	10-150	53-131	40
8270-SIM	3541	Tissue	Chrysene	53-133	56-125	40
8270-SIM	3541	Tissue	Dibenz(a,h)anthracene	31-147	41-144	40
8270-SIM	3541	Tissue	Dibenzofuran	44-118	57-115	40
8270-SIM	3541	Tissue	Dibenzothiophene	25-123	70-130	40
8270-SIM	3541	Tissue	Fluoranthene	45-135	46-132	40
8270-SIM	3541	Tissue	Fluorene	45-121	49-121	40
8270-SIM	3541	Tissue	Indeno(1,2,3-cd)pyrene	33-141	30-148	40
8270-SIM	3541	Tissue	Naphthalene	42-115	28-113	40
8270-SIM	3541	Tissue	Perylene	50-135	70-130	40
8270-SIM	3541	Tissue	Phenanthrene	47-119	54-111	40
8270-SIM	3541	Tissue	Pyrene	50-127	54-112	40
8270-SIM	3541	Tissue	1,2,4-Trichlorobenzene	38-116	59-109	40
8270-SIM	3541	Tissue	1,2-Dichlorobenzene	37-120	51-106	40
8270-SIM	3541	Tissue	1,3-Dichlorobenzene	37-115	43-104	40
8270-SIM	3541	Tissue	1,4-Dichlorobenzene	37-110	45-101	40
8270-SIM	3541	Tissue	2,4,5-Trichlorophenol	22-167	81-132	40
8270-SIM	3541	Tissue	2,4,6-Trichlorophenol	27-161	72-137	40
8270-SIM	3541	Tissue	2,4-Dichlorophenol	51-125	73-140	40
8270-SIM	3541	Tissue	2,4-Dimethylphenol	10-104	56-145	40
8270-SIM	3541	Tissue	2,4-Dinitrophenol	38-172	10-197	40
8270-SIM	3541	Tissue	2,4-Dinitrotoluene	54-133	73-137	40
8270-SIM	3541	Tissue	2,6-Dinitrotoluene	57-124	71-138	40
8270-SIM	3541	Tissue	2-Chloronaphthalene	38-131	37-137	40
8270-SIM	3541	Tissue	2-Chlorophenol	46-118	71-115	40
8270-SIM	3541	Tissue	2-Methyl-4,6-dinitrophenol	48-155	49-148	40
8270-SIM	3541	Tissue	2-Methylphenol	47-111	66-123	40
8270-SIM	3541	Tissue	2-Nitroaniline	50-117	62-131	40

SEMIVOLATILE ORGANICS ANALYSES							
Method	Prep Method	Matrix	Analyte		LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8270-SIM	3541	Tissue	2-Nitrophenol		48-122	52-163	40
8270-SIM	3541	Tissue	3,3'-Dichlorobenzidine		42-129	70-130	40
8270-SIM	3541	Tissue	3-Nitroaniline		53-114	10-127	40
8270-SIM	3541	Tissue	4-Bromophenyl Phenyl Ether		57-122	66-119	40
8270-SIM	3541	Tissue	4-Chloro-3-methylphenol		58-123	68-161	40
8270-SIM	3541	Tissue	4-Chloroaniline		41-93	10-105	40
8270-SIM	3541	Tissue	4-Chlorophenyl Phenyl Ether		59-114	63-123	40
8270-SIM	3541	Tissue	4-Methylphenol		51-110	60-134	40
8270-SIM	3541	Tissue	4-Nitroaniline		52-129	10-129	40
8270-SIM	3541	Tissue	4-Nitrophenol		46-141	49-161	40
8270-SIM	3541	Tissue	Acetophenone		10-137	70-130	40
8270-SIM	3541	Tissue	Aniline		15-74	70-130	40
8270-SIM	3541	Tissue	Atrazine		48-143	70-130	40
8270-SIM	3541	Tissue	Azobenzene		13-143	70-130	40
8270-SIM	3541	Tissue	Benzaldehyde		10-113	70-130	40
8270-SIM	3541	Tissue	Benzoic Acid		10-234	10-215	40
8270-SIM	3541	Tissue	Benzyl Alcohol		33-115	46-133	40
8270-SIM	3541	Tissue	Bis(2-chloroethoxy)methane		47-114	56-124	40
8270-SIM	3541	Tissue	Bis(2-chloroethyl) Ether		52-108	56-112	40
8270-SIM	3541	Tissue	Bis(2-chloroisopropyl) Ether		48-108	42-118	40
8270-SIM	3541	Tissue	Bis(2-ethylhexyl) Phthalate		40-170	53-172	40
8270-SIM	3541	Tissue	Butyl Benzyl Phthalate		59-139	39-171	40
8270-SIM	3541	Tissue	Caprolactam		31-156	70-130	40
8270-SIM	3541	Tissue	Di-n-butyl Phthalate		65-135	47-161	40
8270-SIM	3541	Tissue	Di-n-octyl Phthalate		51-150	35-184	40
8270-SIM	3541	Tissue	Diethyl Phthalate		59-132	60-139	40
8270-SIM	3541	Tissue	Dimethyl Phthalate		62-117	68-124	40
8270-SIM	3541	Tissue	Hexachlorobenzene		58-124	62-121	40
8270-SIM	3541	Tissue	Hexachlorobutadiene		51-105	43-114	40
8270-SIM	3541	Tissue	Hexachlorocyclopentadiene		27-122	10-105	40
8270-SIM	3541	Tissue	Hexachloroethane		50-110	29-116	40
8270-SIM	3541	Tissue	Isophorone		54-122	61-147	40
8270-SIM	3541	Tissue	N-Nitrosodi-n-propylamine		47-121	48-139	40
8270-SIM	3541	Tissue	N-Nitrosodimethylamine		23-113	55-111	40
8270-SIM	3541	Tissue	N-Nitrosodiphenylamine		63-129	71-132	40
8270-SIM	3541	Tissue	Nitrobenzene		48-112	54-119	40
8270-SIM	3541	Tissue	Pentachlorophenol		27-167	10-181	40
8270-SIM	3541	Tissue	Phenol		50-119	55-130	40
8270-SIM	3541	Tissue	Pyridine		12-90	70-130	40
8270-SIM	3541	Tissue	2,4,6-Tribromophenol (Surr.)	47-152	NA	NA	NA
8270-SIM	3541	Tissue	2-Fluorobiphenyl (Surr.)	43-133	NA	NA	NA
8270-SIM	3541	Tissue	2-Fluorophenol (Surr.)	41-112	NA	NA	NA
8270-SIM	3541	Tissue	Fluoranthene-d10 (Surr.)	47-108	NA	NA	NA
8270-SIM	3541	Tissue	Fluorene-d10 (Surr.)	40-96	NA	NA	NA
8270-SIM	3541	Tissue	Nitrobenzene-d5 (Surr.)	35-128	NA	NA	NA
8270-SIM	3541	Tissue	Phenol-d6 (Surr.)	43-133	NA	NA	NA
8270-SIM	3541	Tissue	Terphenyl-d14 (Surr.)	45-139	NA	NA	NA